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AFAPL-TR-68-61

ALKALI METAL RESISTANT ELECTRICAL DEVICES

W. H. Snavely
R. E. Stapleton
A. J. Krause
Westinghouse Electric Corporation

TECHNICAL REPORT AFAPL-TR-68-61

June 1968

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Air Force Aero Propulsion Laboratory
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio



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FOREWORD

This final report is submitted by the Aerospace Electrical Division, Westinghouse Electric Corporation, Lima, Ohio, on Air Force Contract AF33(615)-3528, Budget No. (BPSN) 6(638128-62405214), Alkali Metal Resistant Electrical Devices. The contract was administered by the Air Force Aero Propulsion Laboratory, Aero Space Power Division, Wright-Patterson Air Force Base, Ohio. Mr. Lester Schott was Project Engineer for APJE on this contract.

The work described in this report was done by personnel in the Materials Development, Applied Research and Development Groups of Westinghouse Aerospace Electrical Division, Lima, Ohio.

This report covers work conducted from June 1966 through February 1968. The participation of the following engineers and scientists is specifically acknowledged: A.J. Krause - Test Engineering and Mechanical Design, M.W. Hagadorn and R.E. McVay - Metallurgical Studies, J.W. Ogden - Transformer Design, N.K. Harpster - Circuit Design, and R.E. Stapleton - Ceramic Technology. W.H. Snavely was the Project Engineer and R.M. Frost was the Program Manager.

This report was submitted by the authors for review in May 1968 with contractors designation WAED 68.13E.

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

PHILIP E. STOVER
Act'g Chief, Propulsion
& Power Branch
Aerospace Power Division

ABSTRACT

71. This report covers the evaluation of electrical device materials for 600°C saturated potassium vapor operation. Conductors, insulators, and magnetic materials were exposed to 600°C saturated potassium vapor for various time periods through 5000 hours. Materials were given tests and examinations in the unexposed and exposed conditions, and were evaluated on the basis of this information. Containers for continuously monitoring the electrical resistance of conductors and the d-c magnetic properties of magnetic materials were designed, fabricated, and tested. Processes and techniques were developed for the fabrication of potassium vapor resistant ceramic and/or metal electrical device components. Two 5-kVA transformers were designed, built, and tested to investigate the effects of combined heat, potassium, and electrical and magnetic fields.

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SECTION I

INTRODUCTION

Present design concepts of electrical power generation systems for aerospace and other applications utilize conversion of the thermal energy from lightweight nuclear reactors to electrical energy. Alkali metals are efficient working fluids to accomplish this conversion through turbine alternators. Direct conversion devices also use alkali metals to transfer thermal energy. Electrical devices such as transformers, alternators and capacitors operating in this elevated temperature environment will utilize liquid alkali metal coolants to transfer the excess thermal energy to a suitable radiator. Although various sealing systems to prevent alkali metal vapors from coming into direct contact with the electrical materials are being developed, the possibility of leaks in such seals makes it necessary to use electrical materials that have good corrosion resistance to alkali metal vapor at temperatures to 600°C.

This program was initiated to evaluate electrical materials such as conductors, insulators, and magnetic materials for their corrosion resistance and general performance in 600°C potassium vapor. The test specimens were chosen from materials that were found to be resistant to corrosion by 600°C potassium vapor for short time exposure on previous Air Force contracts.

The objectives of this program were to evaluate candidate materials after 5000 hours exposure to 600°C potassium vapor and use selected conductors, insulators and magnetic materials to fabricate a 5-kVA transformer. The transformer was to be energized in ionized and non-ionized potassium vapor and the performance of the materials and transformer in this environment evaluated.

SECTION II

SUMMARY OF WORK PERFORMED AND MAJOR RESULTS

Magnetic materials, electrical conductors, and insulation materials were exposed to 600°C saturated potassium vapor for time periods through 5000 hours. Material specimens were evaluated by comparing mechanical properties, microstructures, and elemental solution in potassium before and after exposure. Nickel and Inconel clad silver, rhodium, and iridium conductors; Hiperco 27 alloy and Cubex alloy magnetic materials; and hot pressed alumina-yttria eutectic composition, Coors AD999, beryllia, and hot pressed yttria ceramics were either unaffected or little affected by the maximum time exposure to 600°C potassium vapor. Devices to monitor the performance of electrical conductors and magnetic materials in potassium vapor were designed, fabricated, and tested. Electrical resistance of a conductor in 600°C potassium vapor was measured at time intervals for a total time of 900 hours. D-C magnetic tests were the only type of magnetic test that could be performed in boxes containing potassium vapor above a temperature of 238°C due to potassium effecting an electrical short circuit around the core box ceramic insulator. Core loss of Hiperco 27 alloy was determined up to this 238°C temperature in saturated potassium vapor.

Processes and techniques for fabricating materials useful in potassium vapor resistant electrical components were developed and evaluated. Evaluations were made based on the mechanical and/or electrical tests before and after exposure to 600° or 850°C saturated potassium vapor for 200 hours. Hot pressing techniques were developed to fabricate potassium vapor resistant ceramics (alumina-yttria eutectic composition and yttria) and a composite of alumina and alumina-rhodium cermet. Sputtering and plasma-arc spraying techniques were developed to apply potassium vapor resistant interlaminar insulations. Alumina from aluminum oxychloride was used to provide a potassium vapor resistant binder to stiffen a single-crystal whisker sapphire mat.

Two 5-kVA transformers were designed, fabricated, and operated to determine the effects of combined 600°C saturated potassium vapor (both with and without intentional ionization), electric fields, and magnetic fields on materials and electrical operation. Transformers and their instrumented test containers were built of potassium vapor resistant materials and were given electrical tests both with and without potassium and at temperatures in the range from 23° through 600°C. Potassium ionization was found to occur above 180 volts ac.

SECTION III

EXPERIMENTAL WORK

This section describes the experimental work conducted on the program in three general areas.

A. EVALUATION OF ELECTRICAL DEVICE MATERIALS IN POTASSIUM VAPOR

Electrical conductors, insulations, and magnetic materials potentially capable of being used in high-temperature potassium vapor cooled electrical devices were exposed to 600°C potassium vapor for various times through 5000 hours. Table I gives materials tested, capsule material and capsule atmosphere, and test durations. Thermal-vacuum specimens of conductors and magnetic materials were exposed in stainless steel capsules containing no potassium. These specimens were used as control specimens having the same thermal exposure, but without potassium. Capsules containing potassium were sealed to serve as blank standards without specimens. These capsules were given long term tests as standards for potassium impurity pickup from capsules. Other capsules were prepared without specimens for analysis of the starting potassium.

Systems for determining electrical resistance and magnetic properties were designed, fabricated, and tested. These systems made possible the continuous measurement of properties while test materials were exposed to potassium vapor.

1. General Discussion of Items Related to Testing in Potassium Vapor

Several items were common to different tasks in this program. These include equipment used in parts fabrication and potassium loading, test enclosures, processes and techniques associated with potassium and test enclosure handling, and thermal testing. Discussions of these items follow and are referred to throughout the remainder of this report.

a. Inert Atmosphere Glove Box System

An inert atmosphere glove box system was used extensively throughout this program. Its main function was to provide a moisture and oxygen-free argon atmosphere for the operations of potassium handling and tungsten-inert-gas (TIG) welding. Figure 1 shows the inert atmosphere glove box system.

TABLE I. Long-Term Potassium Vapor Exposure Test and Control Specimens

Specimen	Capsule Material	Capsule Atmosphere	600°C Test Duration (hours)			
			1000	2000	3000	5000
Fused Strontium Zirconate	347 Stainless Steel	Potassium	0	0	0	0
Calcined Strontium Zirconate From Chemically Pure Powders	347 Stainless Steel	Potassium	0	0	0	0
Alite A-610	347 Stainless Steel	Potassium	0	0	0	0
Beryllia	347 Stainless Steel	Potassium	0	0	0	0
Coors AD999	347 Stainless Steel	Potassium	0	0	0	0
Felted Sapphire Mat	347 Stainless Steel	Potassium	0	0	0	--
Felted Sapphire Mat With Alumina from Aluminum Oxychloride	347 Stainless Steel	Potassium	--	--	--	0
Alumina-Yttria Eutectic	347 Stainless Steel	Potassium	--	--	0	0
Yttria	347 Stainless Steel	Potassium	0	--	--	0
Boron Nitride Wool	Cb-18 Zr	Potassium	--	--	--	0
Hiperco 27 Alloy	Cb-18 Zr Stainless Steel	Potassium Vacuum	0 0	0 0	0 0	0 0
Cubex Alloy	Cb-18 Zr Stainless Steel	Potassium Vacuum	0 0	0 0	0 0	0 0
Nickel Clad Silver	Cb-18 Zr Stainless Steel	Potassium Vacuum	0 0	0 0	0 0	0 0
Inconel Clad Silver	Cb-18 Zr Stainless Steel	Potassium Vacuum	0 0	0 0	0 0	0 0
Rhodium	Cb-18 Zr Stainless Steel	Potassium Vacuum	0 0	0 0	0 0	0 0
Iridium	Cb-18 Zr Stainless Steel	Potassium Vacuum	0 0	0 0	-- 0	0 0
Hot Potassium Standards	Cb-18 Zr Stainless Steel	Potassium Potassium	0 0	0 0	0 0	0 0



Figure 1. Inert Atmosphere Glove Box System

The glove box was equipped with an antechamber (shown in figure 1 on the right end of the box) and an evacuation subsystem located under the box. This evacuation subsystem consisted of a mechanical roughing pump, an oil diffusion pump, and a liquid nitrogen baffle. Glove box atmosphere was maintained as high purity argon by circulating the gas through a purification subsystem. This subsystem was a modified Vac Atmosphere "DriTrain". Atmosphere purity was verified by monitoring gas from the glove box for moisture with a CEC Moisture Monitor and for free oxygen with a Westinghouse Oxygen Gage. These instruments are shown in figure 1. Most glove box operations were performed with the combined moisture and oxygen contents less than 5 ppm.

Glove box accessories included a subsystem for evacuating containers and test devices inside and independent of the glove box. Inside the glove box were valves connected to flexible lines which were in turn connected to the test devices. Vacuum pumps were outside the glove box. Two roughing pump accessory systems were used to start the main pump, a 140-liter/second ion pump. One roughing system consisted of a manifold of three Varian "Vac Sorb" (liquid nitrogen cooled adsorption) pumps. The other roughing systems consisted of a mechanical pump, a diffusion pump, and a liquid nitrogen trap.

Another glove box accessory system was used to crush and crimp evacuation tubes of containers and test devices. This system consisted of a hand operated hydraulic pump, connecting lines, and a fixture inside the glove box. The fixture consisted of a hydraulic cylinder sealed against oil leakage in a frame with a fixed anvil, and a removable anvil which fitted against the ram of the hydraulic cylinder.

b. Exposure Capsules

Exposure capsules were used to contain potassium while exposing specimens of test materials to potassium vapor. Requirements for capsule materials were that they be resistant to potassium and that they contain a minimum of elements common to the test materials. This latter requirement was imposed so that elements from the exposure specimens could be more accurately evaluated, if present in the potassium after the test was complete.

Exposure capsule designs are shown in figure 2. Capsules were made of 347 stainless steel (used for long term tests of ceramic materials), or columbium-1% zirconium

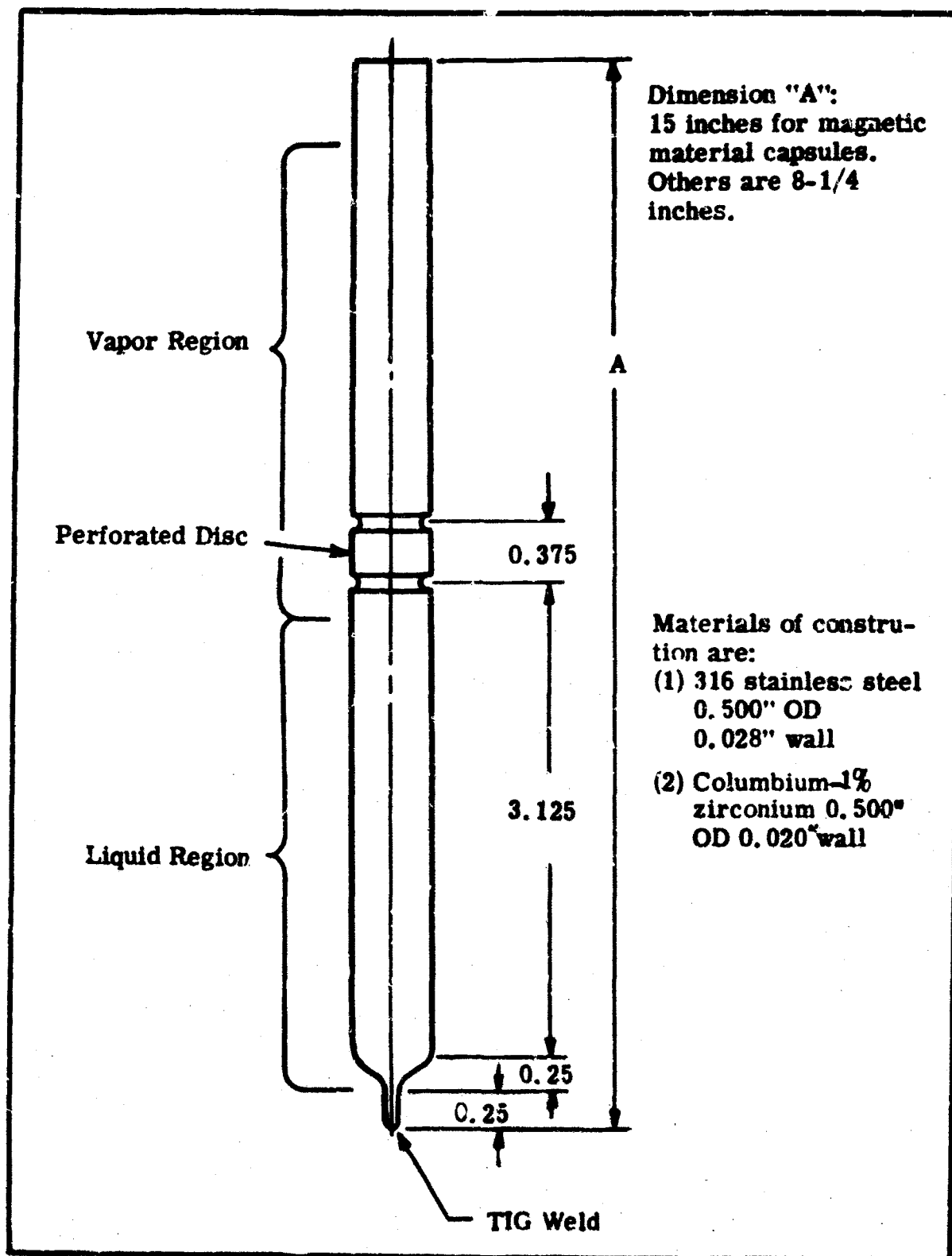


Figure 2. Exposure Capsule for Potassium Vapor Exposure Tests

(used for long term exposures of metallic materials). These materials have demonstrated potassium resistance^{1*}. Table II gives the chemical analysis of the stainless steel tube material. Table III gives chemical analysis of columbium-1% zirconium materials at various stages of forming. The oxygen content of the drawn 0.500-inch outside diameter columbium-1% zirconium tube was less than the specified maximum of 250 ppm.

Exposure capsules of the two materials were processed in different ways. Stainless steel capsule tubing was cut to capsule length, and pre-cleaned according to the following schedule:

- 1) Ultrasonically cleaned in water-detergent solution
- 2) Rinsed in distilled water
- 3) Rinsed in petroleum ether
- 4) Rinsed in acetone
- 5) Dried in an oven at 150°F for 60 minutes

Subsequent to pre-cleaning, the ends of the tubes were crimped and closed by TIG welding. Each weld was then tested for leaks, using a helium leak detector and all were found to be leak tight.

Capsules were then recleaned according to the following:

- 1) Pickled for 2 hours in 10:1 H_2SO_4 - HNO_3 at 80°C.
- 2) Rinsed in distilled water.
- 3) Rinsed in chemically pure acetone.
- 4) Oven dried (150°F for 60 minutes).

Stainless steel capsules were assembled with the addition of a clean 0.030-inch-thick perforated disc to serve as a specimen support above the liquid potassium. These discs had approximately 50% open area with 0.075-inch diameter holes spaced on 0.100-inch staggered centers. Discs had a diameter larger than the capsule inside diameter thus giving a tight fit. Discs were located in the capsule by a groove around the capsule above the liquid potassium reservoir. After assembly, stainless steel capsules were degassed as follows:

- 1) Capsules were placed in a vacuum furnace, and the furnace evacuated to 10^{-6} torr.
- 2) Capsules were degassed at 1100°F for 8 hours.

* See references.

TABLE II. Stainless Steel Tube Chemical Analysis

Chemical Analysis (suppliers analysis)	
Element	Percent Content
C	0.051
Mn	1.60
P	0.016
S	0.011
Si	0.63
Cr	17.84
Ni	11.99
Cu	0.13
Cb/Ta	0.87
Mo	0.35
NOTE: Stainless steel tubing meets specifications MIL-T-8808A and AMS 5571B. Heat No. 28979. 1/2-inch OD x 0.028-inch-thick wall.	

- 3) Capsules were cooled in vacuum, then the furnace was back-filled with high purity dry argon.
- 4) The furnace was opened and the capsules were transferred to the argon filled glove box.
- 5) Swagelok caps were installed on capsules while in the glove box, thus sealing them in high purity argon.
- 6) Sealed capsules were then placed in transport containers, removed from the glove box, and stored in transport containers under 40 psi argon. The argon pressure was used to assure O-ring sealing. Figure 3 shows one such capsule.

TABLE III. Columbium-1% Zirconium Chemical Analysis

Heat Analysis (Suppliers Analysis)		
Percent		
	Tcp	Bottom
Zr	0.80	0.82
Cb	Balance	
Impurity Content, ppm		
Al	<20	<20
B	<1	<1
C	50	40
Cd	<5	<5
Co	<10	<10
Cr	<20	<20
Cu	<40	<40
Fe	<50	<50
H	3.8	4.6
Hf	<80	<80
Mg	<20	<20
Mn	<20	<20
Mo	35	35
N	35	35
Ni	<20	<20
O	<50	<50
Pb	<20	<20
Si	<50	<50
Sn	<10	<10
Ta	<500	<500
Ti	<40	<40
V	<20	<20
W	70	80
Product chemistry of 0.625 inch OD by 0.065 inch ID tube, ppm		
C		<30
N		48
O		120
H		3.6
Product chemistry of 0.500 inch OD by 0.020 inch wall tube, ppm		
O		228

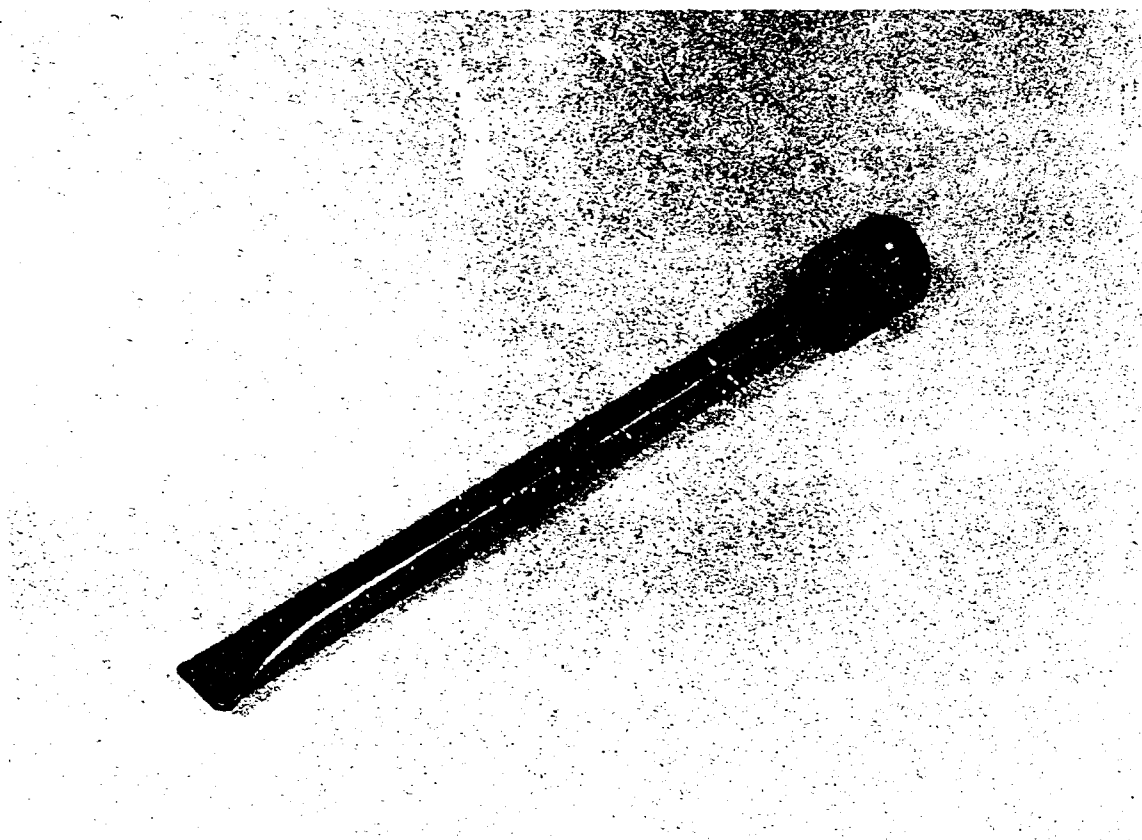


Figure 3. Stainless Steel Exposure Capsule Prior to Potassium Loading

Columbium-1% zirconium capsules were processed according to the following:

- 1) Tubing was cut to length.
- 2) Tubes were cleaned in petroleum ether.
- 3) Tubes were rinsed in chemically pure acetone.
- 4) Tubes were oven dried at 200°F for 30 minutes.
- 5) Tube ends were pinched closed giving approximately 3/16-inch flat length.
- 6) Pinched ends were recleaned with chemically pure acetone and air dried at 200°F for 30 minutes.
- 7) Tubes were placed in the inert atmosphere glove box vestibule and the vestibule was evacuated.
- 8) Tubes were transferred to the inert atmosphere glove box.
- 9) Tube pinched ends were TIG welded in an argon atmosphere. Combined oxygen and moisture content of the argon atmosphere was maintained below 0.3 ppm.
- 10) Capsules were helium leak tested and found to be leak free.
- 11) Perforated discs of columbium-1% zirconium with the same perforations as the stainless steel discs were prepared. These discs had a diameter of 29/64 inches.
- 12) Capsules were grooved, the perforated discs were installed, and a second groove placed above the discs thus securing them in place.
- 13) Completed capsules were then vacuum annealed and degassed by heating to 2200°F in a vacuum of 10^{-8} torr and maintaining this temperature for one hour.
- 14) Capsules were furnace cooled to room temperature and transferred to the glove box.
- 15) Swagelok caps were installed on capsules while in the glove box, thus sealing in high purity argon.
- 16) Sealed capsules were then placed in transport containers, removed from the glove box, and stored in transport containers under 40 psig argon.

c. MSA Potassium Loading

Long term potassium vapor exposure capsules were loaded with potassium at MSA Research Corporation, Evans City, Pennsylvania. Loading potassium directly from the supplier's container into the capsule minimized the possibility of contamination in potassium transfer.

Cleaned and sealed capsules were shipped to the potassium supplier (MSA) in transport containers pressurized with high purity argon. Containers were made of three inch schedule 40 type 316 stainless steel pipe with a 0.375-inch plate of the same material welded to form a bottom. The top was of type 316 stainless steel with an O-ring

for sealing and was held in place by three studs which were welded to the pipe.

Each container top had two valves. One valve was used for container evacuation; the other to introduce a protective gas atmosphere. Figure 4 shows examples of these containers. Transport containers were opened in MSA's high purity argon atmosphere glove box which was equipped for potassium loading.

Effluent argon from the glove box was continuously monitored during loading and was found to contain less than one ppm oxygen and less than one ppm moisture during all loadings. Potassium from the supplier's container was analyzed for oxygen, carbon, and metallic impurities. Table IV gives the MSA Research Corporation analysis of potassium loaded into most long term potassium vapor exposure test capsules. This table shows that the analyzed sample contained less than ten ppm oxygen.

Capsule loading began with removing the Swagelok cap which sealed in high purity argon. A hypodermic needle was then placed in the capsule extending through the perforated plate which separated the potassium vapor and liquid regions. A potassium level sensing probe was inserted into the capsule so its shoulder rested on the perforated plate and the sensing tip extended into the liquid potassium region. The capsule and needle were heated with a heat lamp to a temperature above that at which potassium melts. A valve on the heated potassium container in the glove box was opened and liquid potassium, under pressure of high purity argon, flowed into the capsule until potassium contacted the probe. This contact completed a sensing circuit thus signaling that the potassium supply valve should be closed.

The potassium height sensing probe was made of 0.125-inch diameter columbium-1% zirconium rod with the ends reduced to 0.050-inch diameter to fit through holes in the plates. Figure 5 shows two of these probes. Either end of the rods could be used for potassium sensing. Probe insulation consisted of plasma arc sprayed high purity alumina (99.99%). Alumina was selected as the insulation because of its abrasion and potassium resistance. Columbium-1% zirconium was selected for its potassium resistance and expansion compatibility with the insulation.

Upon completion of the potassium loading, capsules were resealed with Swagelok caps and placed in transport containers. When the transport containers were loaded, the

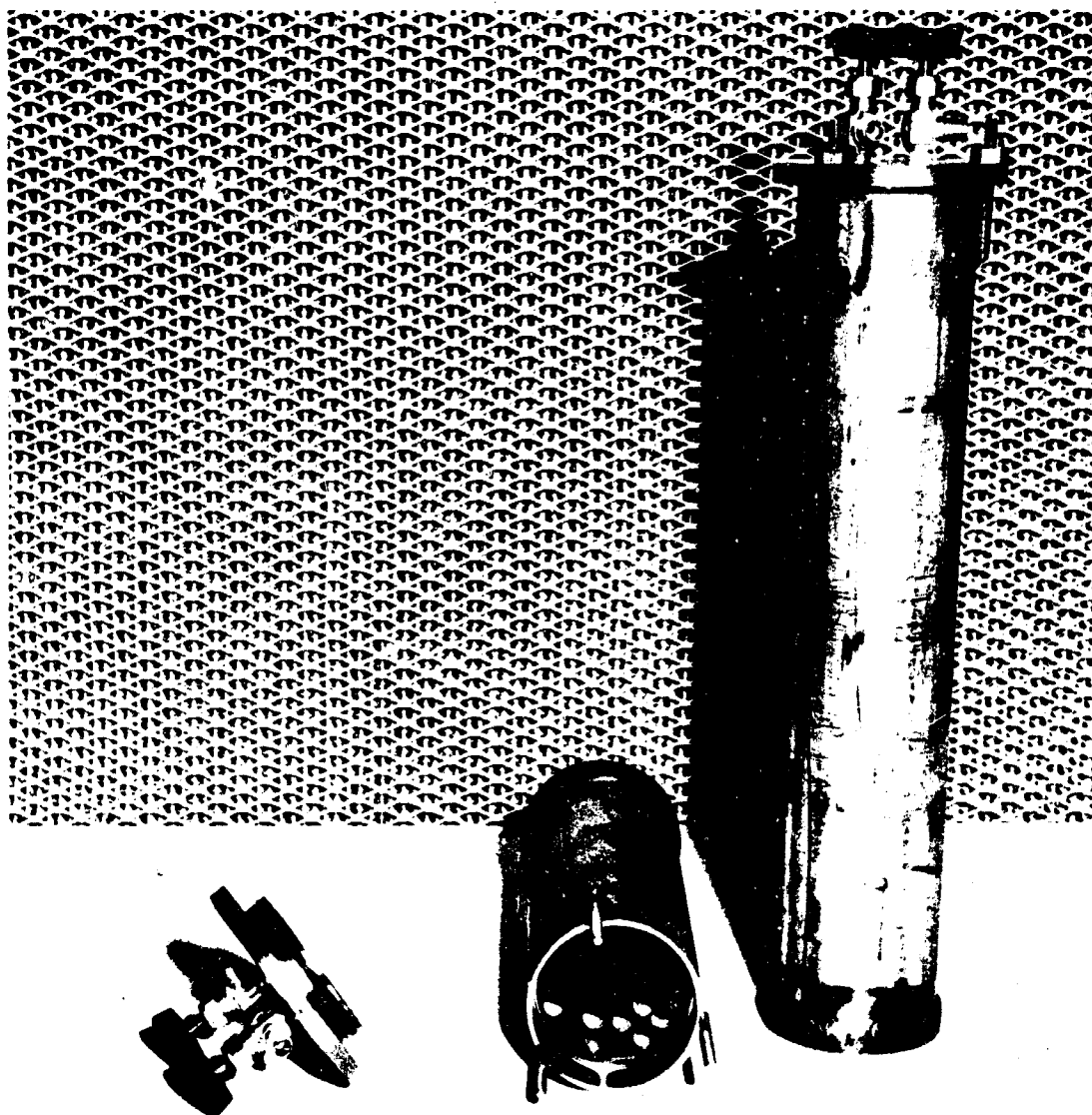


Figure 4. Assembled and Disassembled Transport Containers

TABLE IV. Potassium Chemical Composition

Supplier: MSA Research Corp., Evans City, Pa.	
Potassium Lot Number ST7-857-1	
Batch Analysis, Metallic Impurities, (ppm)	
Fe	<5
B	<10
Co	<5
Mn	<1
Al	<2
Mg	<2
Sn	<2
Cu	<2
Pb	<5
Cr	<5
Si	12
Ti	<5
Ni	<5
Mo	<3
V	<1
Be	<1
Ag	<1
Zr	<1
Sr	<1
Ba	<3
Ca	1
Na	4
K	Bal.
Container Analysis, (ppm)	
O ₂	<10
C	10

tops were put on, the valves closed, and they were removed from the glove box. Once outside the glove box, the transport containers were pressurized to 40 psig with high purity argon and checked for leaks. They were then transported and received for specimen loading and final sealing.

d. Westinghouse Potassium Loading

Potassium for other than long term potassium vapor exposures was purchased from MSA Research in glass ampoules. Table V gives the batch impurity analysis of this potassium. An analysis of the potassium,



Figure 5. Potassium Liquid Level Probes

TABLE V. Ampoule Potassium Impurity Analysis

High Purity Potassium Lot No. ST7-857-1	
Element	Batch Analysis (ppm)
Fe	<5
B	<10
Co	<5
Mn	<1
Al	<2
Mg	<2
Sn	<5
Cu	2
Pb	<5
Cr	<5
Si	12
Ti	<5
Ni	<5
Mo	<3
V	<1
Be	<1
Ag	<1
Zr	<10
Sr	<1
Ba	<3
Ca	1
Na	4
K	Balance
An ampoule loaded at the same time as those received was analyzed for oxygen and was found to contain less than 10 ppm oxygen	

loaded at the same time as the ampoules, showed the oxygen content to be less than ten ppm.

Potassium loading at Westinghouse was by hypodermic syringe. The syringe and needle were prepared by chemical cleaning (rinsing in chemically pure acetone) then vacuum degassed (10^{-6} torr range) at 400°F for a minimum of two hours. The main body of the syringe and the needle were electrically heated by a resistance heater wire electrically insulated with woven silica sleeving. The syringe plunger was heated during degassing on a laboratory hot plate. After degassing and the introduction of purified argon to the glove box, the syringe was assembled and the syringe and hot plate temperature controllers set to maintain 200°F. A scored ampoule was opened and placed in a holding fixture on the hot plate. After the potassium had melted, it was transferred by the heated syringe and injected into the test device. The quantity of transferred potassium was controlled volumetrically by using a calibrated syringe.

e. Exposure Capsule Specimen Loading, Evacuation, and Sealing

Cleaned specimens, capsules, and associated loading equipment were placed in the glove box and the glove box evacuated. MSA loaded capsules were introduced to the glove box in their pressurized transport containers. Other capsules were placed on the hot plate for vacuum degassing. Specimens were placed on the hot plate and were degassed at 400°F in the evacuated glove box. After suitable evacuation the glove box was back-filled with high purity argon and the argon was circulated through a purification system until the oxygen and moisture levels were both less than one part per million. Specimens were allowed to cool during the back-fill. Transport containers were opened, capsules removed, capsules opened, specimens inserted, and the capsules evacuated. In the case of thermal-vacuum exposure, specimens were placed in capsules containing no potassium. The same procedures were used for capsules which were standards for potassium analysis except no specimens were included.

The capsule evacuation line was pumped to less than 5×10^{-6} torr, as indicated at the ion pump, before sealing. Evacuated capsules were crimped and both cut off and sealed using TIG welding. The crimp was held by hydraulic pressure during sealing to assure that low pressure was maintained inside the capsule. Residual

gas was analyzed for representative capsule loadings to determine the approximate level of water, oxygen, and other compounds. At a total pressure of 5×10^{-7} torr (measured at the ion pump), the partial pressure of hydrogen was approximately 1×10^{-7} torr and that of water vapor was approximately 4×10^{-7} torr.

f. Capsule Container Loading and Thermal Exposure

Capsules to be exposed for a given time duration were placed together in cleaned containers. Titanium sponge was added to act as a getter for surface oxygen from the container. Container tops were then welded in place and the weld was checked for leaks. Loaded containers were evacuated, crimped, and sealed by TIG welding as were the capsules. Sealed containers were placed in air furnaces for either 600° or 850°C exposure. Typical capsule containers are shown in figure 6.

g. Vacuum Furnace For Potassium Vapor Device Exposure

Test containers for transformers, electrical conductors, Hiperco 27 alloy Rowland rings, and a sapphire window electrical terminal test container all used columbium-1% zirconium in potassium vapor resistant ceramic-to-metal seals. These containers were tested in a hot-wall vacuum furnace to assure an atmosphere free of contaminants, such as oxygen, which would attack the columbium-1% zirconium at test temperatures. The vacuum portion of the furnace consisted of a mechanical roughing pump, an oil diffusion pump, and a liquid nitrogen cooled baffle. The furnace consisted of a stainless steel cylinder and end plates with radiation shields inside. The center portion of the cylinder was electrically heated by a helical winding. One furnace end plate was equipped with vacuum tight feedthroughs for electrical power, thermocouples, and a window for viewing test containers.

h. Capsule Cooling and Unloading

Exposure capsule containers were cooled after thermal testing using one of the two different methods. The first method used was to place the hot test container on a fire brick and allow it to be cooled by room air convection. Potassium was found to be in the upper or specimen regions of these capsules. When potassium was to be recovered for impurity analysis (i.e. from long term tests) additional steps were required to

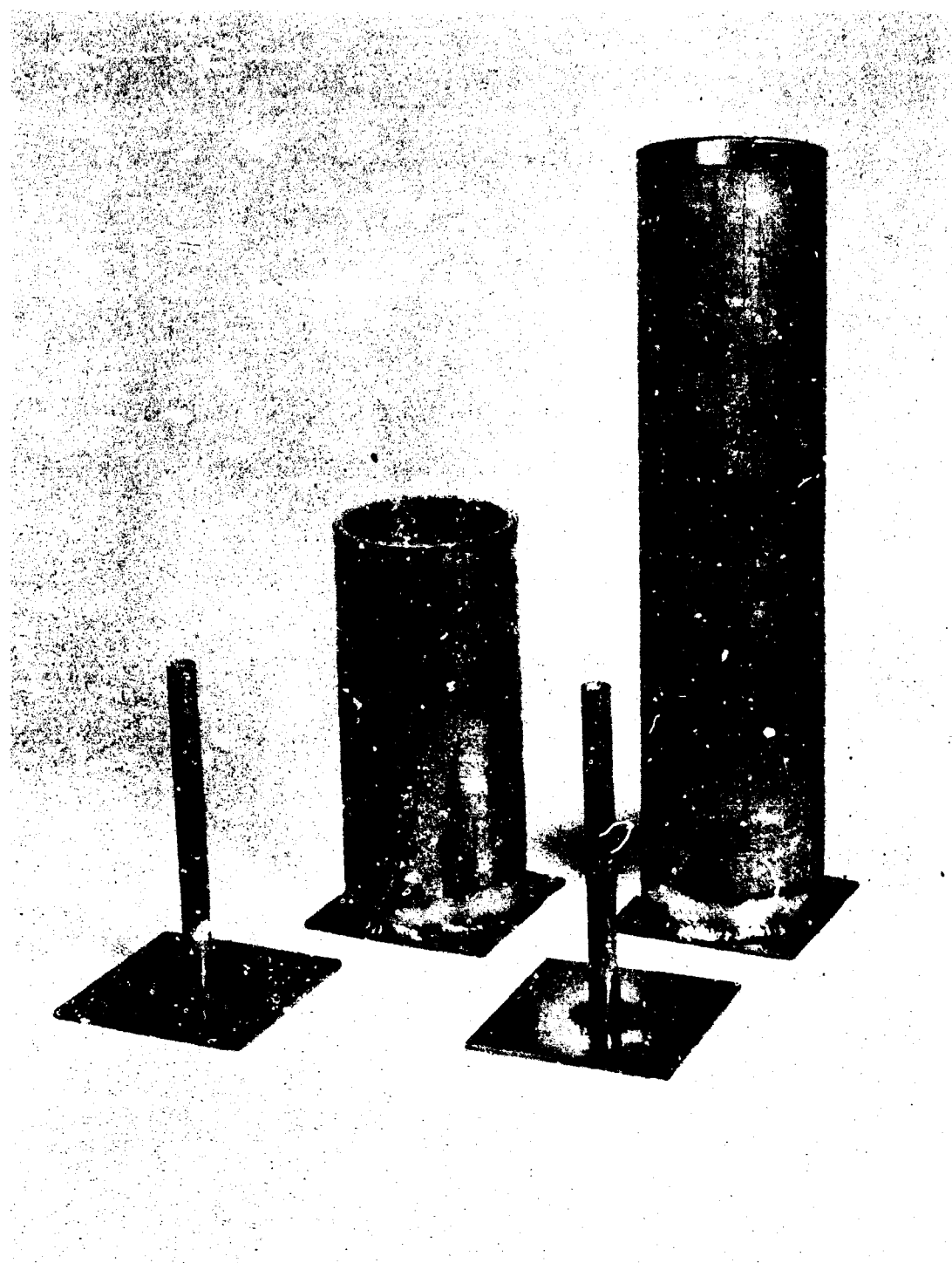


Figure 6. Stainless Steel Containers for Potassium Vapor Exposure Capsules

separate the potassium from the specimens. The second method used for capsule cooling was first to place the lower end of the test container in water and then gradually raise the water level up the container. This latter cooling method condensed all of the potassium in the capsule's lower region, except for thin films which adhered to the capsule walls, to and between specimens, and between specimens and capsule walls.

Opening of the test container began by sawing off the fill and evacuation tubes, thus allowing air to enter. The crimped end of the fill tube was then examined for evidence of potassium. If potassium was found, (thus indicating leaking capsules) the container was flushed with methyl alcohol under a cover of flowing argon followed by flushing with water. This neutralized and removed the potassium. Container top plates were then cut off and exposure capsules removed.

Exposure capsules containing potassium were unloaded using one of two methods if the potassium was to be analyzed for impurities. When potassium was not to be analyzed, exposure capsules were opened at Westinghouse Electric Corporation (WAED) in a glove box with an argon cover flowing into the capsule. This glove box was vented so that hydrogen (resulting from neutralizing potassium) and argon were exhausted. Potassium was neutralized by reacting it with methanol. After the potassium was neutralized, specimens were removed from capsules, washed with methanol, washed with distilled water, oven dried at 100+°C, then cooled and stored in a desiccator before further processing.

Capsules containing potassium from long term tests (both hot standards and specimen-containing) and the potassium cold standards were taken to Technical Services Laboratory, Westinghouse Atomic Power Division, for unloading and potassium analysis. Capsule exteriors were cleaned with acetone as the first step in the potassium unloading sequence. Cleaned capsules were then placed in an inert atmosphere glove box vestibule where evacuation and back filling were performed prior to passing the capsules into the glove box. Capsules were handled in the glove box in a high purity helium atmosphere. During the times when potassium was exposed to the glove box atmosphere the combined oxygen and moisture level was maintained at between 2 and 3 ppm.

Capsules were opened by circumferential cuts made with a tubing cutter. In capsules where potassium had been condensed in the lower region, specimens were removed with the upper capsule portion. When potassium had condensed in the specimen region of the capsule, specimens were removed from their capsules by heating the potassium and pouring the specimens and potassium into glass vessels. Specimens, potassium, and tubes were then transferred to an argon glove box where potassium was reacted with methyl alcohol to form potassium methyllate. Potassium methyllate solution and undissolved impurities were then placed in suitable plastic evaporating beakers in preparation for analyses.

Two observations were made during the opening of capsules containing specimens. First, all specimens had coatings of potassium on their surfaces with the quantity being a function of the cooling cycle. Second, all potassium had a high luster, indicating low oxygen content.

Hot and cold potassium standard capsules were prepared by cleaning as were the capsules containing specimens and were handled singly in the helium atmosphere glove box. Two potassium samples were removed from each capsule for oxygen analysis by cutting off sections of tubing containing potassium. Each sample was reacted with mercury and then processed by standard methods for oxygen determination by combined titration and by flame photometry. The remaining potassium in each capsule was used for metallic impurity analysis.

i. Mechanical Test Equipment

Strength values were determined on unexposed and exposed materials from both long term and short term potassium vapor exposure tests. These tests were performed on an Instron Model PTC tensile testing machine. Metallic specimens were pulled using air pressure actuated steel clamps to grip the specimens. Yield point or 2% offset yield strengths, and elongation values for metallic specimens were determined from stress-strain plots made by this machine. Ceramic modulus-of-rupture specimens were broken using the four-point loading fixture shown schematically in figure 7.

2. Evaluation of Potassium Vapor Exposed Electrical Conductors

Four types of electrical conductors were processed, given potassium and/or thermal exposure, tested and evaluated.

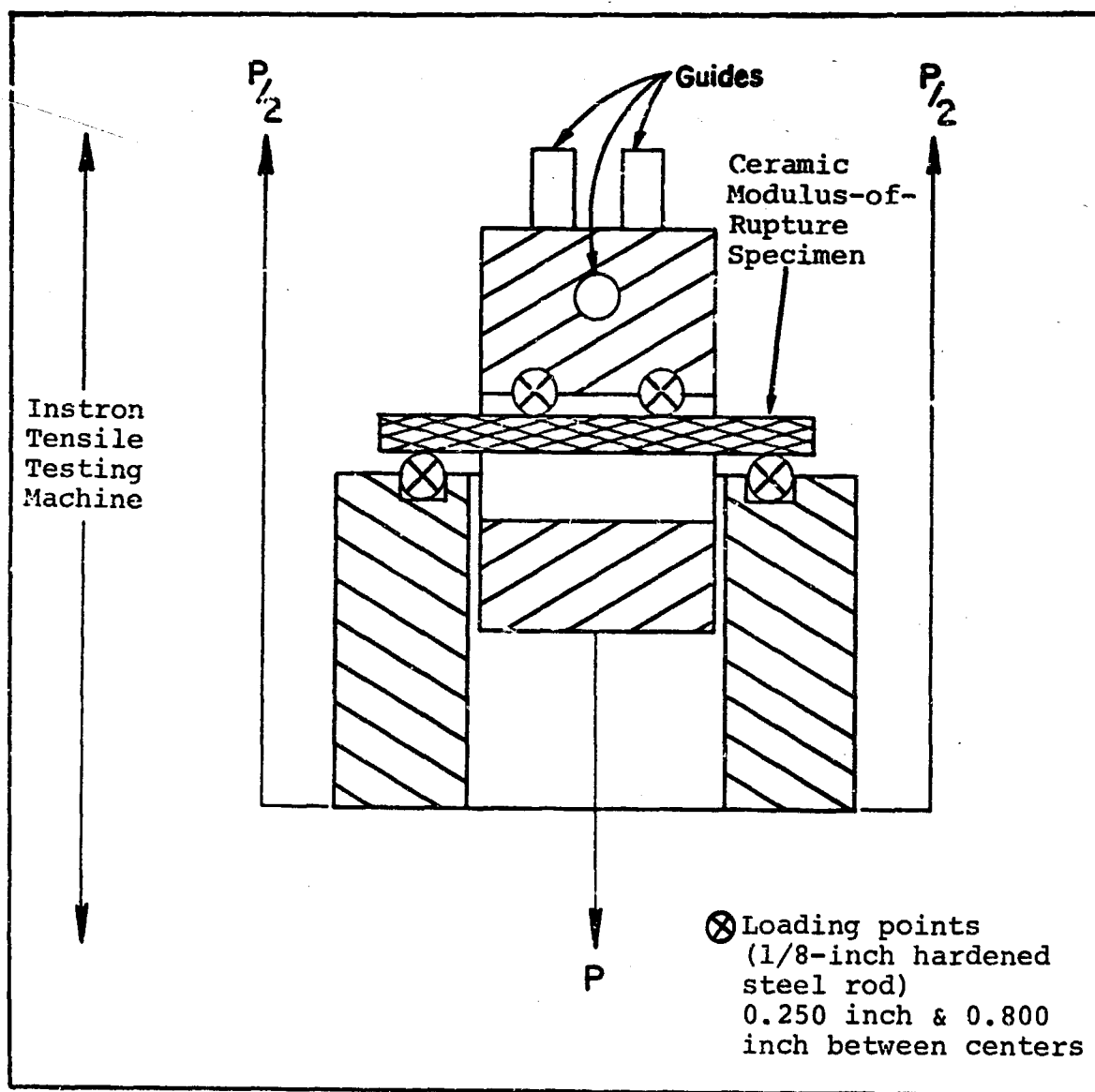


Figure 7. Modulus-of-Rupture Four-Point Loading Fixture

a. Specimen Preparation

Conductors were ordered in the annealed condition from various suppliers. Table VI lists these materials and their suppliers. The nickel-clad silver used in this portion of the program was purchased on an earlier program* as No. 6 and No. 8 circular conductor with 28 volume percent nickel. This material was redrawn by the supplier into a rectangular form approximately 0.081-inch by 0.129-inch with the same cladding proportion. Inconel-clad silver was No. 10 circular wire with 20 volume percent Inconel cladding.

Silver cores of these conductors are not resistant to potassium. Surface preparation and plating processes were developed to protect the silver cores. Both wire types were first ultrasonically cleaned in methyl chloroform to remove oil and other soluble surface contaminants, then acid etched. Nickel-clad silver was etched for one minute in a 180°F solution containing one part hydrochloric acid and two parts water. Wires were then washed with water to remove acid. Inconel-clad silver was prepared for plating by a 20 to 30 second etch in a solution containing two gallons water, one gallon nitric acid, and one pint hydrofluoric acid. After etching, these wires were neutralized in a 1 to 2% ammonium hydroxide solution, then rinsed with water.

TABLE VI. Potassium Vapor Exposed Electrical Conductors

Test Material	Source
Iridium Rhodium	Englehard Ind., Newark, N.J. Sigmund Cohn Corp., Mt. Vernon, N.J.
Nickel-Clad Silver	Sylvania Parts Div., Warren, Pa.
Inconel-Clad Silver	Sylvania Parts Div., Warren, Pa.

* Contract No. AF33(615)-1360

Both conductor types were nickel plated using parameters and solutions as follows:

Temperature	130° F
pH range	3.5 to 5.0
Agitation	Controlled speed stirrer
Anode	99+% rolled and depolarized nickel.
Voltage	6 to 9 volts
Current Density	50 to 100 amps per square foot
Plating Solution from Barrett Chemical Products Division.	
Allied Composition	
Nickel sulfamate	60.00 ozs/gal
(Nickel metal content)	(10.20 ozs/gal)
Boric Acid	5 ozs/gal
Anti-pit agent (SNAP)	0.05 oz/gal

Both types of clad conductor specimens were eight inches long. Figure 8 shows specimens of clad wire before and after plating.

Both rhodium and iridium were ordered as 0.030-inch diameter annealed wire. The suppliers reported the minimum purity as rhodium-99.95% and iridium-99.8%. Iridium was obtained in two lots, both from the same supplier. Spectrographic analyses of rhodium and iridium are given in table VII. Specimen preparation consisted of cutting to a 6-inch length, bending a 1/8-inch "el" on one end to keep the specimen from falling through the exposure capsule disc perforations, and cleaning with chemically pure acetone. A series of tensile tests was performed on unexposed iridium wires of both 4- and 8-inch lengths. All breaks were in the gage length (i.e. not in the jaws). Tensile strengths were not affected by specimen length.

b. Electrical Resistivity Tests

Electrical resistivities of conductors were determined as a function of temperature. All measurements except those at 18.9°C were made in vacuum. Wires were tested in the "as-received" condition. The standard ASTM method was used with a specimen gage length of 30 cm and resistances measured with a Kelvin bridge. Resistivities were calculated from resistance measurements and conductor areas.

Resistivities of rhodium are given in figure 9. The Englehard² referenced data point appears to be compatible with both curves. Melpar data on Englehard material shows lower resistivity than Westinghouse data on Cohn material. Differences in resistivity of these suppliers' materials have been noted before³.

The resistivity of iridium as a function of temperature is shown in figure 10. Iridium was a better electrical conductor than rhodium at temperatures above 150°C (see figure 9).

c. Evaluation of Mechanical Tests

Mechanical tests were performed on conductor materials. These tests gave values for actual yield point or yield strength determined at 0.2% offset and percent elongation in two inches for both thermal-vacuum and thermal-potassium exposed specimens. These values were calculated from measured parameters as discussed in Appendix I.

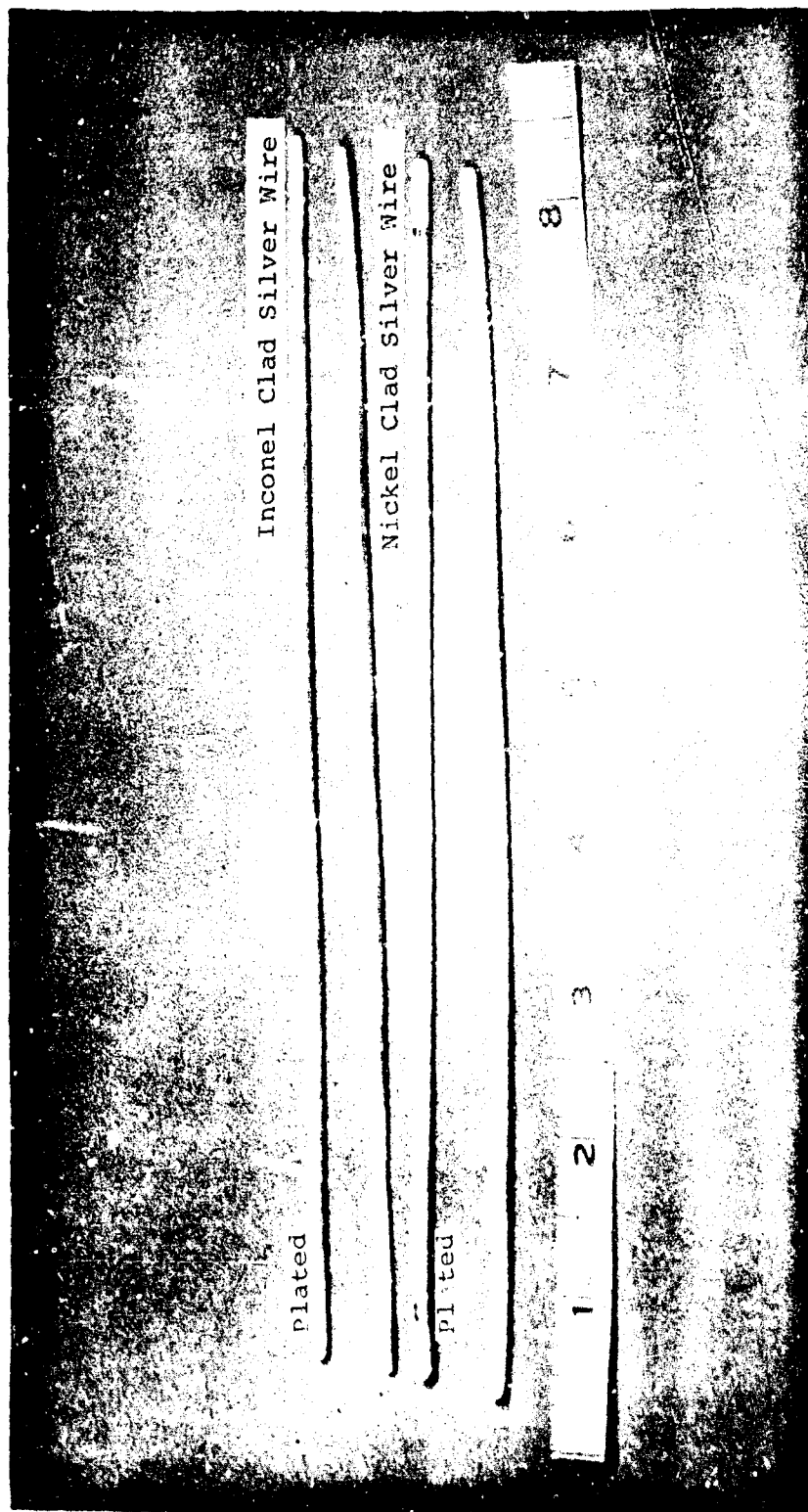


Figure 8. Clad Silver Wire Specimens Before and After Plating

TABLE VII. Spectrographic Analysis of Iridium and Rhodium Wires

Element	Iridium Wire Lot No. 1 (% Trace)	Rhodium Wire (% Trace)
Gold	<0.001	<0.001
Gallium	<0.001	<0.001
Hafnium	0.004	<0.002
Indium	<0.002	<0.002
Iridium	Balance	<0.005
Platinum	0.01	0.001
Rhenium	<0.001	<0.001
Rhodium	0.08	Balance
Ruthenium	<0.002	<0.002
Palladium	0.01	<0.001
Copper	0.0005	0.0004
Nickel	0.0009	0.008
Silver	<0.001	<0.001
Iron	<0.005	<0.005
Manganese	<0.0005	0.0008
Silicon	<0.001	<0.001
Analyses performed by Bowser-Morner Laboratories Dayton, Ohio		

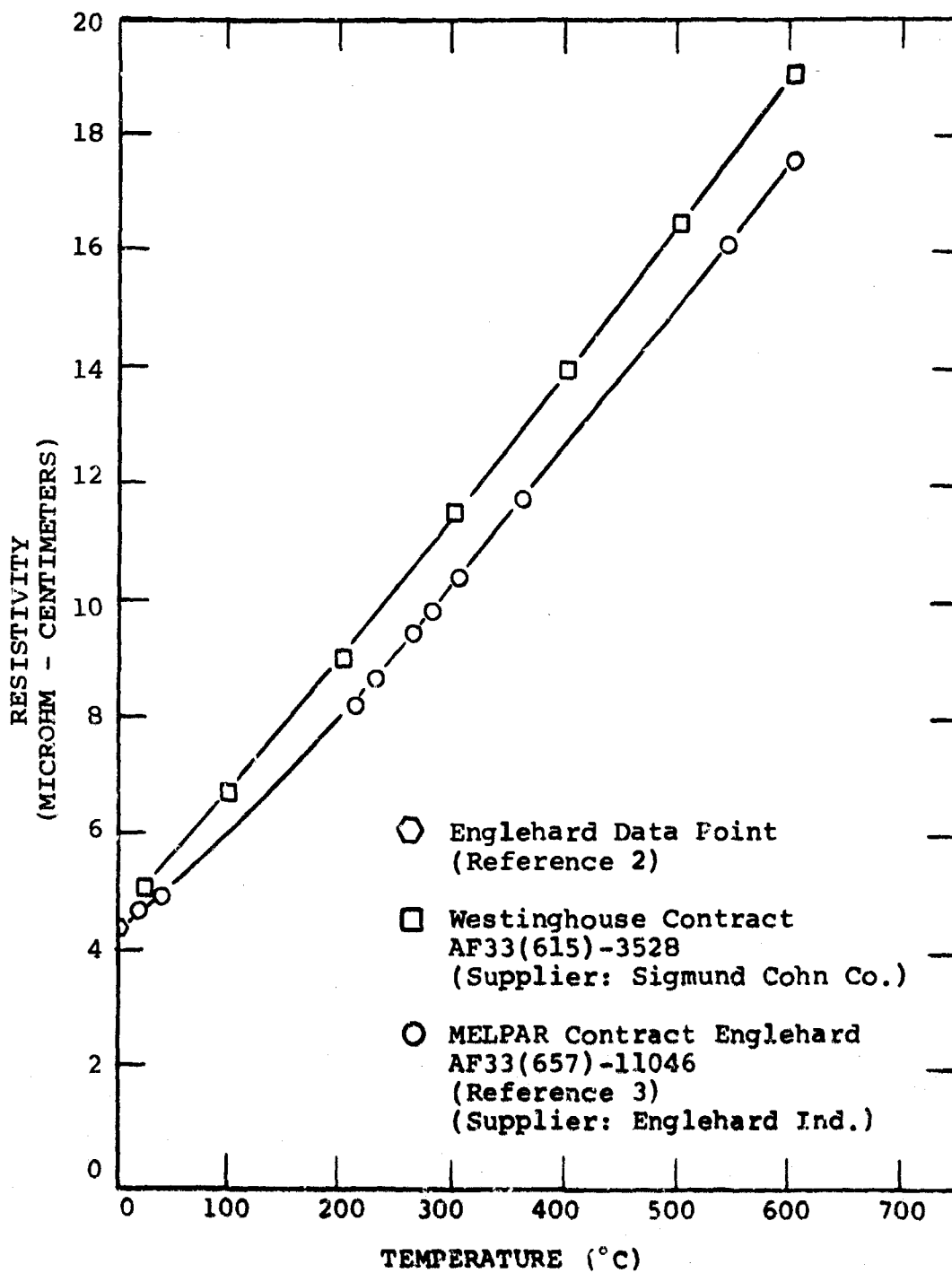


Figure 9. Electrical Resistivity of High Purity Rhodium Wire

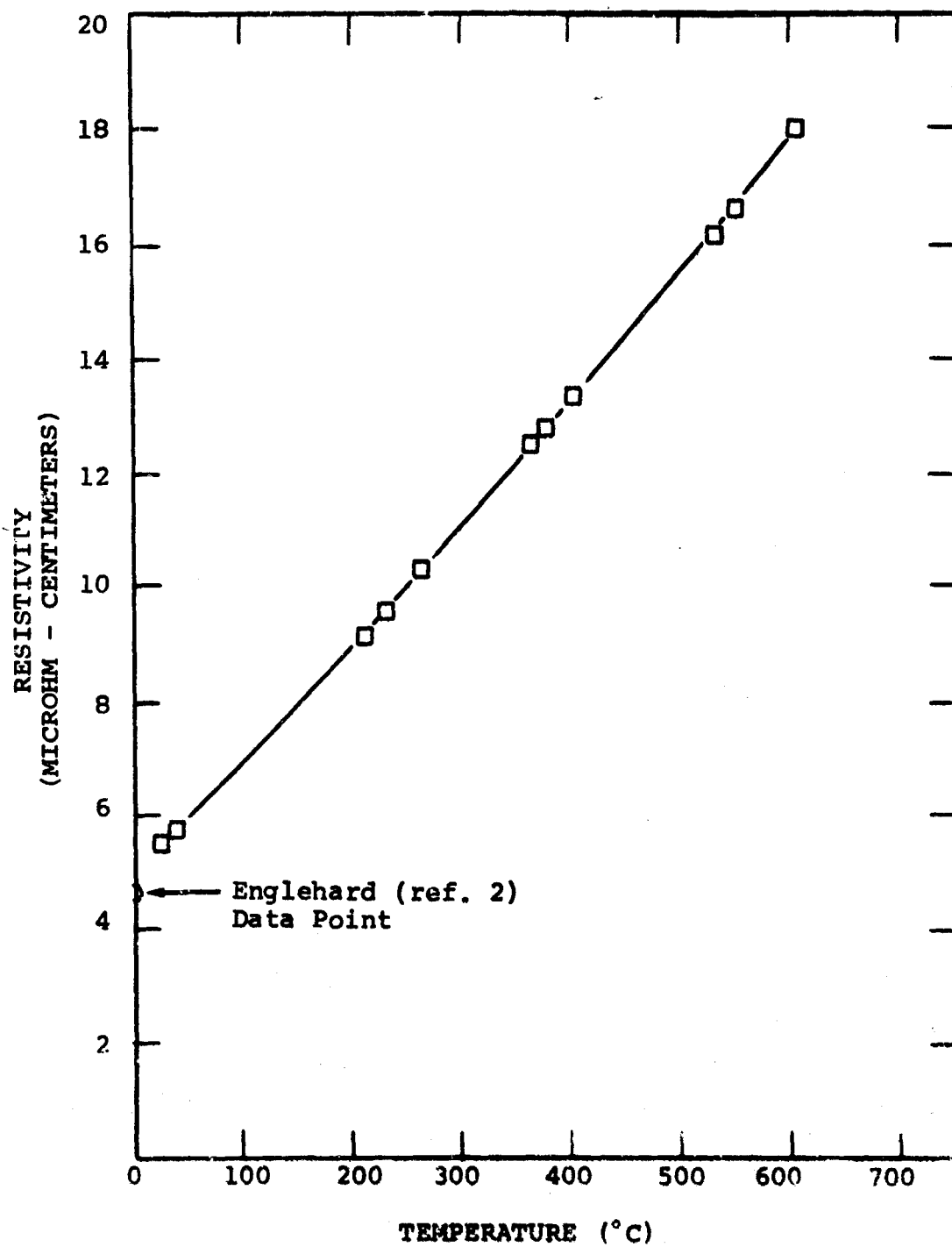


Figure 10. Electrical Resistivity of High Purity Iridium Wire

Individual values were examined and compared with the average value and the standard deviation, also discussed in Appendix II. Values outside the standard deviation were discarded and revised averages were calculated. Table VIII gives the revised mechanical test data and their coefficients of variation. Average values of these data are plotted in figures 11, 12, and 13.

Figure 11 gives average mechanical test data on nickel-clad silver. Yield points are seen to decrease and elongation to increase with exposure time in both the potassium and thermal vacuum tests. The major portion of the changes occurred early in testing (within the first 2000 hours). Changes appear to be due to stress relief annealing which was not complete in the "as-received" or unexposed material. Potassium, itself, appears to have had no adverse effects on nickel-clad silver conductors.

Average mechanical test data for Inconel-clad silver conductors are shown in figure 11. Elongation of this material was little affected by thermal aging with or without potassium. Yield point and yield strength appear to be decreased by the thermal portion of test only. Inconel-clad silver conductors appear to have been unaffected by potassium.

Figure 12 shows averaged mechanical test data for iridium. Iridium 0.2% offset yield strength was unaffected in these tests. The increase in elongation with exposure time and the differences between thermal vacuum and thermal potassium are considered anomalous behavior.

Average mechanical test data for rhodium are shown in figure 13. Yield point and elongation were not affected by exposure. The "as-received" rhodium wire had a unique property in that the wire could be bent once easily but could not withstand a reverse bend. This property created a serious problem in winding resistivity test specimens. The chemical analyses in table VII indicate that the major impurities in the rhodium and iridium wires consist of the platinum group metals.

d. Evaluations of Microstructures

Specimens of conductor materials were obtained prior to mechanical testing. These specimens were mounted, polished, and etched; and given microstructure examinations

both before and after etching. Figures 14 through 17 show the microstructures of conductors after 5000-hour tests. These specimens were etched as is shown in table IX.

Microstructures of the conductor materials were examined in the "as-received" condition, after thermal exposure in 600°C vacuum, and after 600°C potassium vapor exposure. No evidence of potassium attack was found.

Figure 14 shows nickel-clad silver after 5000 hours of vacuum and potassium vapor exposures. An increase in the grain size of the core silver in both the nickel and Inconel-clad specimens occurred between 3000 and 5000 hours. This could have resulted from a temperature escalation during this interval. Figure 15 shows Inconel-clad silver microstructures after both thermal vacuum and potassium vapor exposures for 5000 hours. No evidence of change can be found.

Iridium exhibited a fibrous microstructure and is shown in figure 16 after 5000-hour exposures. The fibrous structure resulted from cold work during wire drawing. No evidence of structure change was found due either to the thermal or potassium exposures. Figure 17 shows the microstructure of rhodium after 5000 hours in potassium vapor and vacuum at 600°C. No evidence of change was found.

e. Evaluation of Impurity Pickup Analyses

Potassium from standards and long term exposure capsules was analyzed for impurities. An increase of a conductor constituent element above its standard impurity level would be a sign of potassium corrosion. Impurity level change with exposure time would give an indication of corrosion rate and could possibly be correlated with any mechanical properties or microstructure changes. Analyses of potassium from standards and specimen capsules is given in table X.

The oxygen content of potassium in capsules containing no specimens (i.e. standards) at the exposure time intervals is also shown in table X. These standards were either stored at room temperature or exposed at 600°C with capsules containing specimens. A comparison of the supplier's (MSA) oxygen analysis of <10 ppm with the oxygen content of the potassium after 1000 to 5000 hours at 600°C shows no appreciable increase in oxygen

TABLE VIII. Mechanical Properties of Unexposed and Long Term Potassium Exposed Electrical Conductors

Material	Unexposed			1000 Hour Exposed			2000 Hour Exposed	
	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)
Nickel Clad Silver								
Vacuum	33,627 35,098 34,607 35,588	34,730	2.0	16,336 19,801 20,495 20,792 20,599 20,297	19,719	7.83	13,225 13,725 13,725 13,529	13,554
Potassium							16,470 15,882 15,980 16,666	16,250
Material	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)
Nickel Clad Silver								
Vacuum	18.5 15 15	16.17	10.20				40 43 46 44	43.25
Potassium							45 43.5 44 41.5	43.5

Material	Unexposed			1000 Hour Exposed			2000 Hour Exposed	
	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)
Inconel Clad Silver								
Vacuum	30,375 31,250 32,250 31,250	31,211	2.12	29,252 28,252 28,872 28,500	28,469	0.89	27,261 27,881 27,261	27,468
Potassium				27,633 27,881 28,004 29,120	28,160	1.03	27,633 27,137 27,261	27,344
Material	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)
Inconel Clad Silver								
Vacuum	18.5 22 21.5 22	21	6.94	24 25 24.5	24.5	1.67	27 28 28 28	27.75
Potassium				22.5 20 24	22.17	7.44	24.5 26	25.25

Unexposed and Long Term Electrical Conductors

0 Hour Exposed		2000 Hour Exposed			3000 Hour Exposed			5000 Hour Exposed		
Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)
13,719	7.83	13,225 13,725 13,725 13,529	13,554	1.3	13,039 13,235 13,235	13,170	0.70	14,901 15,000 15,490	15,130	1.70
		16,470 15,882 15,980 16,666			15,686 15,980 15,393 14,705			16,176 14,116 14,705 15,000		
Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)
		40 43 46 44	43.25	5.0	38 35 35 38	36.5	4.1	40 47 40	40.3	1.17
		45 43.5 44 41.5			44 42.5 47 45			42 43 45 40		
			43.5	2.9		44.67	3.2		42.5	4.2

0 Hour Exposed		2000 Hour Exposed			3000 Hour Exposed			5000 Hour Exposed		
Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)
28,469	0.89	27,261 27,881 27,261	27,468	1.06	28,500 26,393 27,261 26,641	27,199	2.5	29,739 29,120 29,120 29,120	29,274	0.9
		27,633 27,137 27,261			27,881 26,889 26,641			27,881 27,261 27,013		
28,160	2.03		27,344	0.77		27,137	1.97		27,385	1.33
Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)
24.5	1.67	27 26 28 28	27.75	1.6	28 28 24 23	25.75	8.8	24 25 25	24.67	1.91
		24.5 26			26 25 21			23.5 20 21		
22.17	7.44		25.25	2.97		24	9.0		21.5	6.84

TABLE VIII. - Continued

Material	Unexposed			1000 Hour Exposed			2000 Hour Exposed	
	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)
Iridium								
Vacuum	208,571 218,571 205,714 214,285	211,785	2.51	238,571 250,000 250,000 247,142	246,428	1.90	221,428 205,714 182,571	203,238
Potassium				242,857 250,000 254,285 246,571	248,861	1.63	207,142 200,000 214,285	207,142
Material	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)
Iridium								
Vacuum	10 10 9	9.67	4.88	9 8.5 7.5	8.33	7.5	9 10	9.5
Potassium				8.5 7.5 11.0	9.0	16.35	12.5 12.5	12.5

Material	Unexposed			1000 Hour Exposed			2000 Hour Exposed	
	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)
Rhodium								
Vacuum	22,857 19,642 21,071 18,571	20,535	7.82	22,142 25,714 28,571 25,714	26,785	4.42	22,850 20,000 24,285 20,000	21,784
Potassium				25,714 24,285 27,142	25,714	4.54	22,850 22,850 24,285	23,320
Material	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)
Rhodium								
Vacuum	13.2 13 14 13.5	13.4	2.80	12.5 13.5 11	12.33	8.33	19 20 18 20	19.25
Potassium				10 12.5 14	12.17	13.56	20 19 19	19.33

Used	2000 Hour Exposed			3000 Hour Exposed			5000 Hour Exposed		
Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)
1.90	221,428 205,714 182,571	203,238	6.8	204,285 214,285 214,285 221,428	213,570	2.17	175,914 172,857 172,857	173,876	0.82
1.63	207,142 200,000 214,285	207,142	2.8				185,714 178,571 161,428	175,238	5.81
Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)
7.5	9 10	9.5	5.26	9 10 10	9.67	4.88	13 14 12 13	13	5.4
16.35	12.5 12.5	13.5	0				17.5 15 18	16.83	7.79

Used	2000 Hour Exposed			3000 Hour Exposed			5000 Hour Exposed		
Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)
4.42	22,850 20,000 24,285 20,000	21,784	8.2	24,285 25,714 22,850	24,283	4.81	22,857 21,485 20,000	21,447	5.44
4.54	22,850 22,850 24,285	23,328	2.90	27,142 25,714 27,142 25,714	26,428	2.7	28,571 22,850 20,000	23,807	14.97
Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)
8.33	19 20 18 20	19.25	4.3	18 19 20	19	4.30	15 12 15	14	10.10
13.56	20 19 19	19.33	2.44	15 13 13	13.33	9.35	14 12	13	7.69

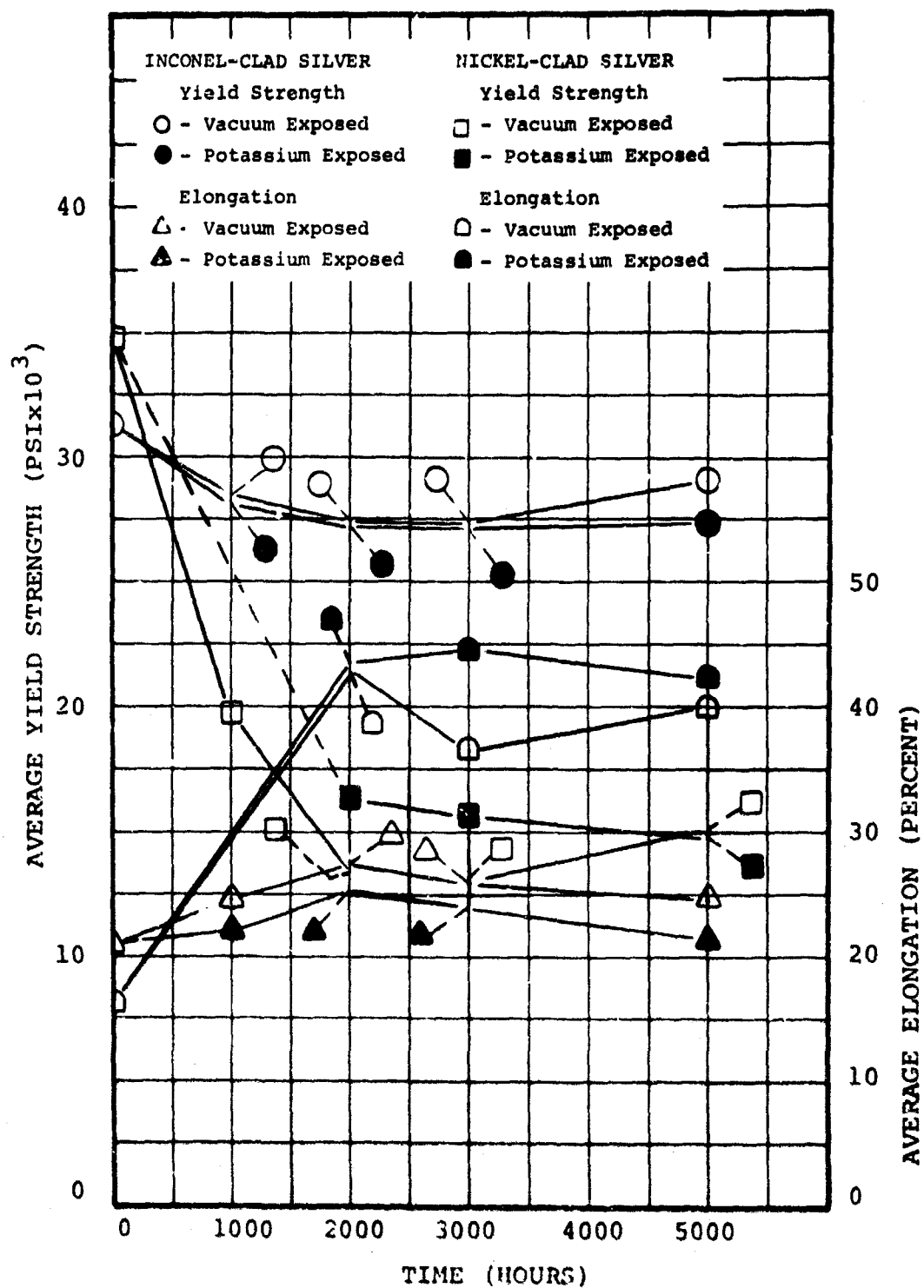


Figure 11. Average Mechanical Properties of Inconel and Nickel Clad Silver Conductors

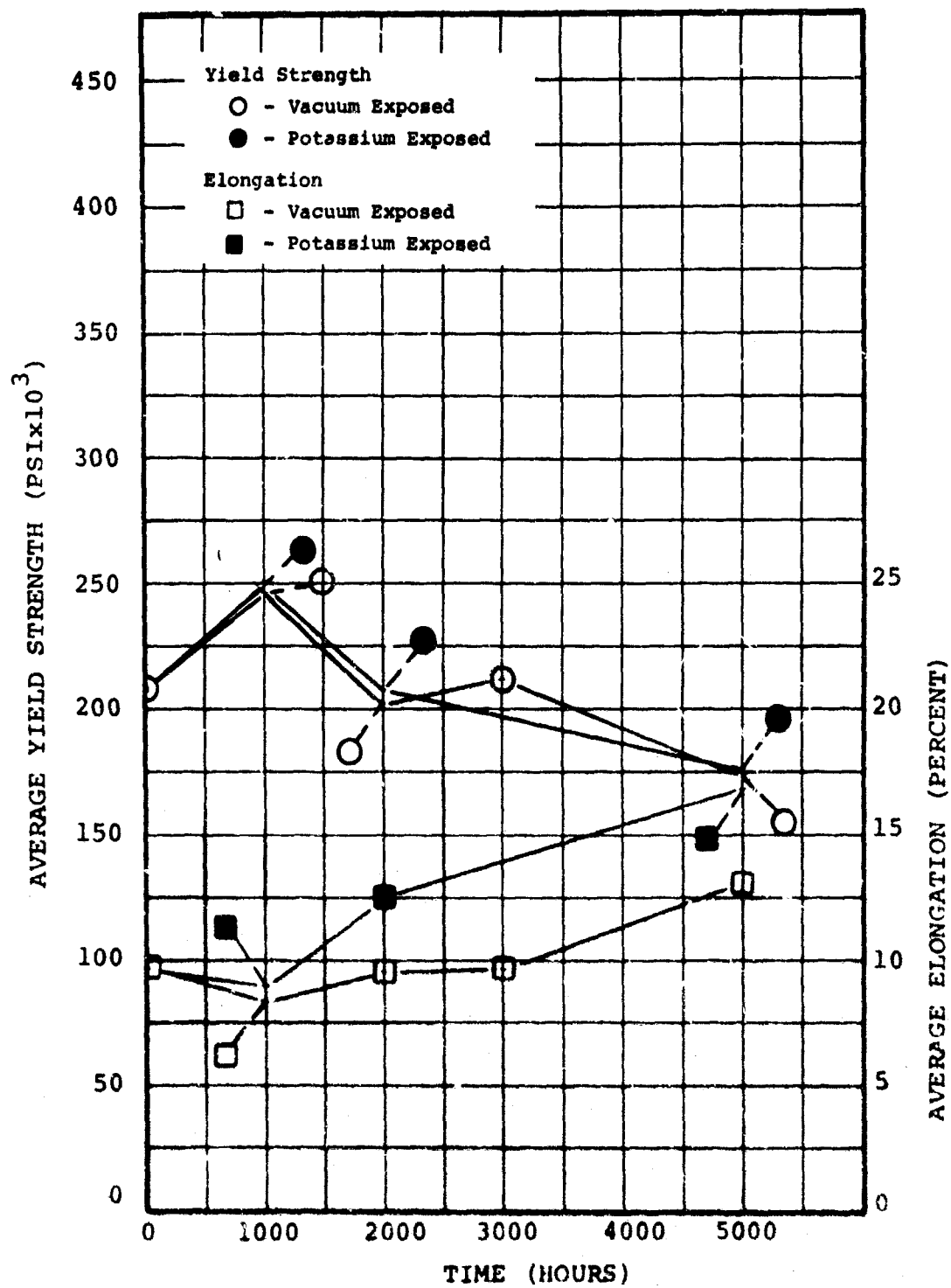


Figure 12. Average Mechanical Properties of Iridium Wire

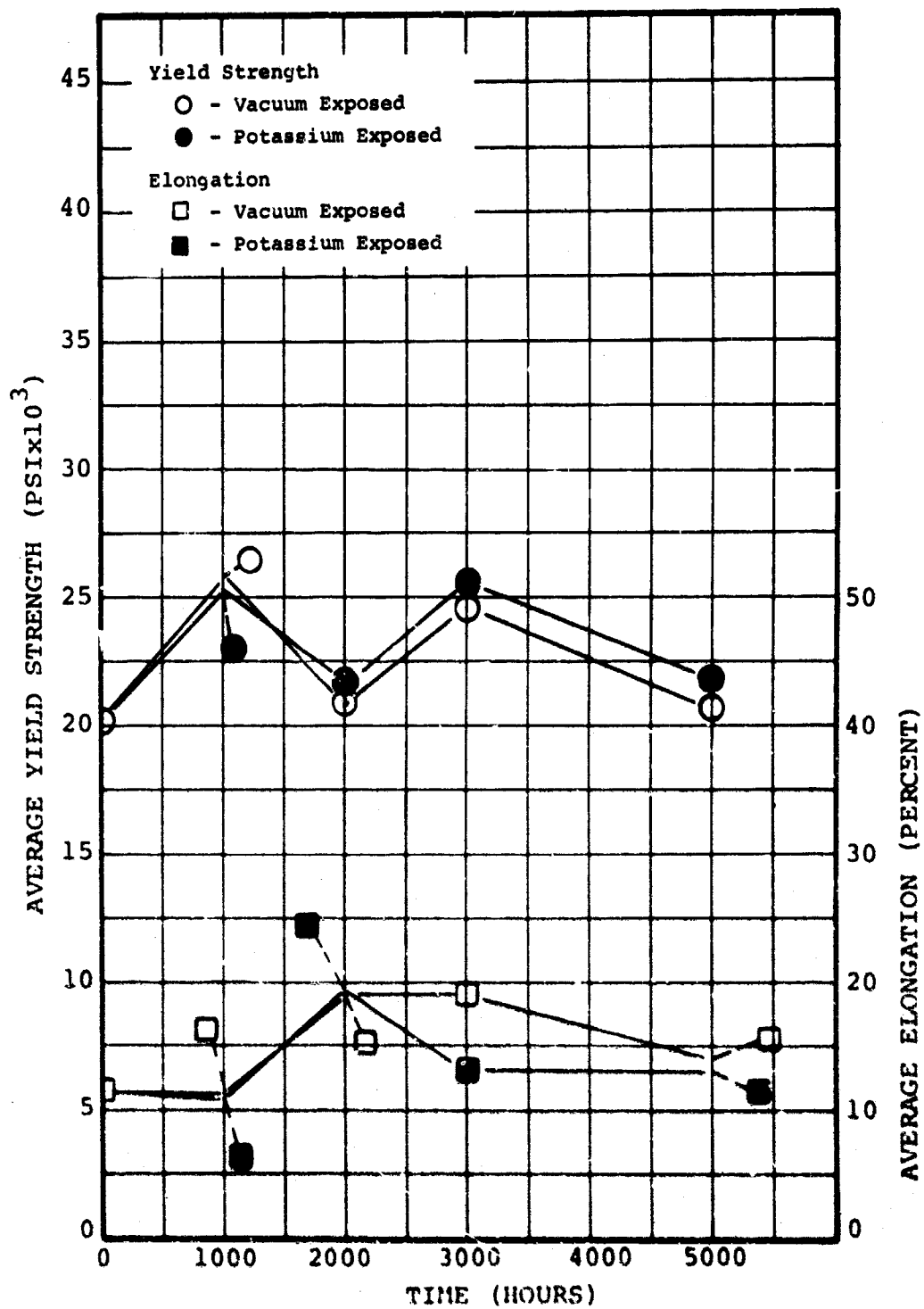


Figure 13. Average Mechanical Properties of Rhodium Wire

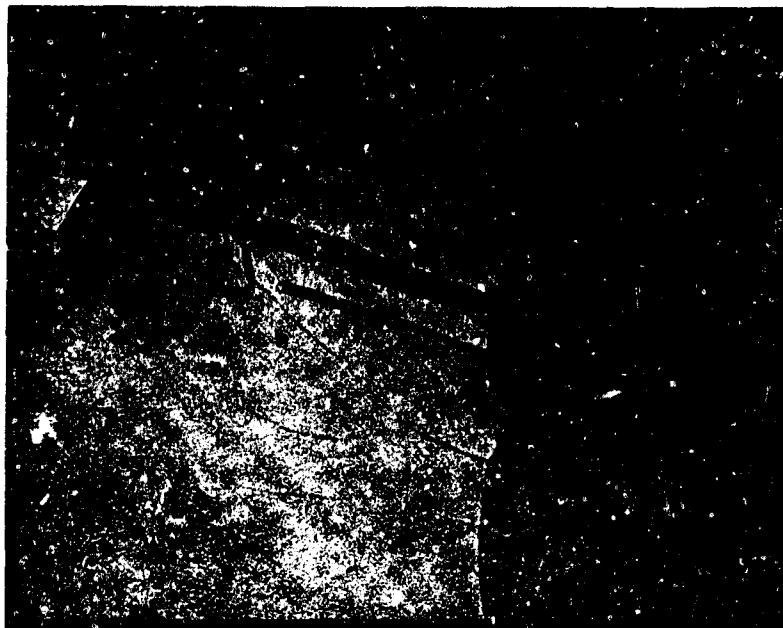


Thermal Vacuum Exposure



Potassium Vapor Exposure

Figure 14. Micrographs of Silver From Nickel-Clad Silver
After 5000 Hours at 600°C Thermal Exposure With
and Without Potassium Vapor (100X)

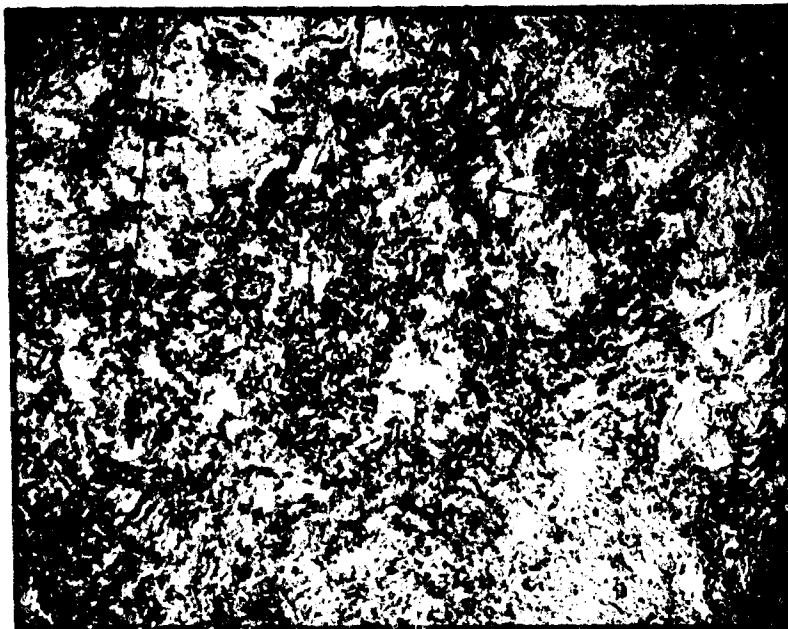


Thermal Vacuum Exposure

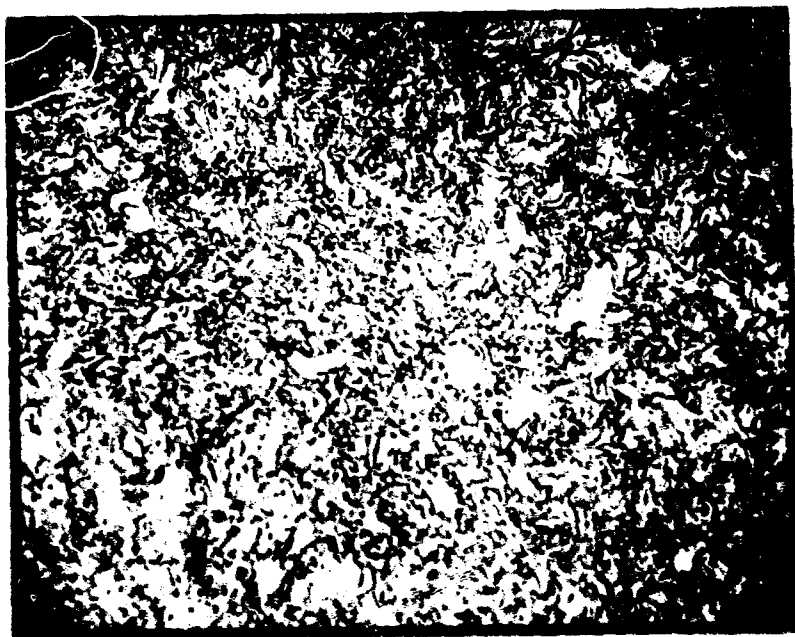


Potassium Vapor Exposure

Figure 15. Micrographs of Silver in Inconel-Clad Silver After 5000 Hours at 600°C Thermal Exposure With and Without Potassium Vapor (100X)

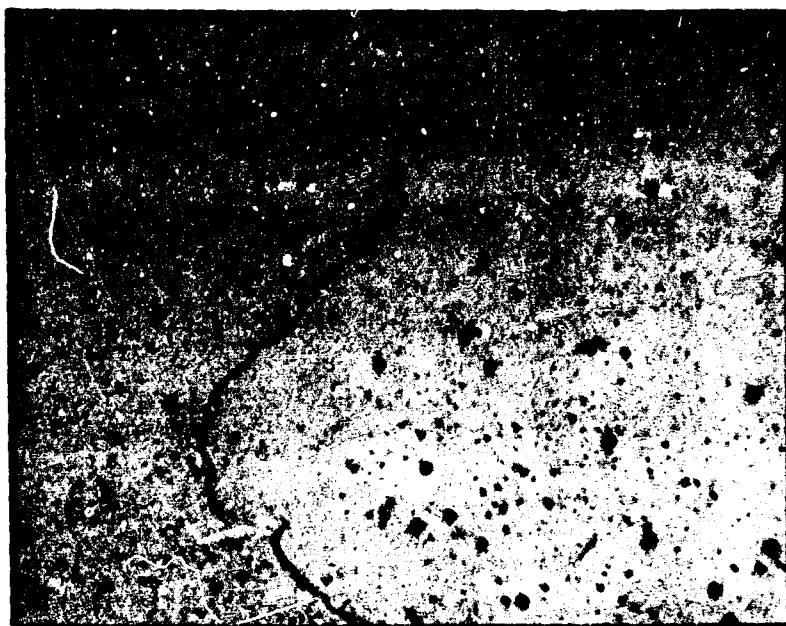


Thermal Vacuum Exposure

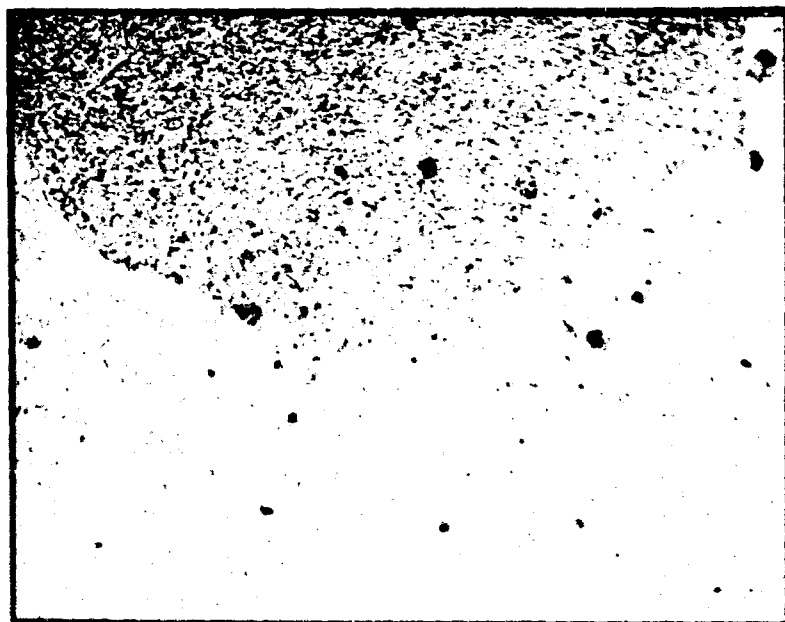


Potassium Vapor Exposure

Figure 16. Micrographs of Iridium After 5000 Hours at 600°C
Thermal Exposure With and Without Potassium Vapor
(160X)



Thermal Vacuum Exposure



Potassium Vapor Exposure

Figure 17. Micrographs of Rhodium After 5000 Hours at 600°C
Thermal Exposure With and Without Potassium Vapor
(800X)

TABLE IX. Etchants Used on Electrical Conductors

Material	Etchant
Nickel-Clad Silver	10% Potassium Cyanide 10% Ammonium Persulfate Solution
Inconel-Clad Silver	10% Potassium Cyanide 10% Ammonium Persulfate Solution
Iridium	20 ml HCl, 90 ml H ₂ O AC current 0.02-0.1 amperes per square centimeter
Rhodium	Conc HCl Saturated with NaCl, 5 volts ac

content except for the value of 69 ppm after 2000 hours.

Potassium from 2000- and 3000-hour exposure nickel-clad silver capsules had considerable amounts of silver. An examination of loaded capsule X-rays showed that specimens had slipped past the support discs and had fallen into the liquid potassium. An examination of the specimens showed the plated nickel that had been in liquid potassium was corroded. Mechanical tests of nickel-clad silver (see figure 11) indicated the potassium did not diffuse into and weaken the test sections. The solubility of silver in potassium is evidently limited with saturation at 3.8% silver for water quench conditions.

The rhodium, iridium, and nickel-clad silver conductor specimens showed no solution in 600°C potassium vapor as there was no appreciable difference between the potassium analysis of the exposure samples and the standards. None of the conductors exposed were attacked by potassium vapor.

TABLE X. Analyses of Potassium in Conductor Specimen Exposure Capsules

Specimen (a)	500°C Exposure (hour)							
		Ag	Al	Au	B	Ba	P ₂	Bi
Iridium	1000	< 0.6	7.2	< 600	< 6	< 6	< 2	< 20
	2000	< 2	< 6	< 100	< 6	< 2	< 0.6	< 6
	3000						No Potass	
	5000	23	11	< 100	< 6	< 6	< 0.6	< 6
Rhodium	1000	< 0.6	15	< 600	< 6	< 6	< 2	< 20
	2000	< 2	< 6	< 100	< 6	< 2	< 0.6	< 6
	3000	< 2	6.0	< 100	< 6	< 2	< 0.6	< 6
	5000	< 2	15	< 100	< 6	< 6	< 2	< 6
20% Nickel- Clad Silver	1000						No Potass	
	2000	38000	760	< 100	5.7	< 2	< 0.6	< 2
	3000	38000	7.6	< 100	< 6	< 2	< 0.6	< 6
	5000	150	19	< 100	< 6	< 2	< 0.6	< 6
20% Inconel- Clad Silver	1000	< 0.6	11	< 600	< 6	< 6	< 2	< 20
	2000	9.3	76	< 100	5.7	< 2	< 0.6	< 2
	3000	150	11	< 100	< 6	< 2	< 0.6	< 6
	5000	15	13	< 100	< 6	< 2	< 0.6	< 6
Blank Standard (Cb-1Zr)	1000	< 0.6	1.9	< 600	< 6	< 6	< 2	< 20
	2000	< 2	9.9	< 100	< 6	< 20	< 20	< 6
	3000	2	48	< 100	< 6	< 2	< 2	< 6
	5000	< 2	32	< 100	< 6	< 2	0.6	< 2
Batch Analysis of Potassium		< 1	< 2		< 10	< 3	< 1	
Blank Standard (Cb-1Zr) 23°C		< 0.6	4.8	< 600	< 6	< 6	< 2	< 20

(a) Conductors were exposed in columbium-1% zirconium capsules

Exposure Capsules

Impurity (ppm)

Ba	Be	Bi	Ca	Cd	Co	Cr	Cu	Fe	Ir	Mg	Mn
< 6	< 2	< 20	< 2	< 60	< 6	1.9	3.1	53	< 200	2.3	20.6
< 2	< 0.6	< 6	5.7	< 6	< 2	13	31	34	< 100	2.3	0.57
No Potassium Was Found In This Capsule											
< 6	< 0.6	< 6	< 6	< 6	< 2	40	6.0	190	< 100	3.1	4.4
< 6	< 2	< 20	< 2	< 60	< 6	19	4.8	55	< 200	2.3	0.92
< 2	< 0.6	< 6	7.6	< 6	< 2	< 6	44	40	< 100	3.2	0.57
< 2	< 0.6	< 6	11	< 6	< 2	< 6	< 6	< 6	< 100	< 0.6	< 0.6
< 6	< 2	< 6	< 6	< 6	< 2	< 6	< 6	13	< 100	3.2	< 0.6
No Potassium Was Found In this Capsule											
< 2	< 0.6	< 2	15	38	< 2	160	76	430	< 100	10	19
< 2	< 0.6	< 6	< 6	6	< 2	< 6	19	32	< 100	0.57	< 0.6
< 2	< 0.6	< 6	< 6	< 6	< 2	< 6	23	51	< 100	4.6	< 0.6
< 6	< 2	< 20	< 2	< 60	< 6	0.57	2.5	5.7	< 200	1.8	< 0.6
< 2	< 0.6	< 2	19	340	< 2	270	67	610	< 100	10	21
< 2	< 0.6	< 6	15	11	< 2	27	< 6	170	< 100	3.2	2.9
< 2	< 0.6	< 6	< 6	< 6	< 2	< 6	< 6	17	< 100	1.4	< 0.6
< 6	< 2	< 20	5.7	< 60	< 6	4.8	5.8	170	< 200	0.95	1.5
< 20	< 20	< 6	3.8	< 20	< 6	9.5	23	34	< 100	< 0.6	1.2
< 2	< 2	< 6	2	< 20	< 6	48	290	270	< 100	7.6	10
< 2	0.6	< 2	< 2	< 6	< 2	19	16	23	< 100	< 0.6	< 0.6
< 3	1		1		< 5	< 5	2	< 5		< 2	< 1
< 6	< 2	< 20	5.7	< 60	< 6	4.8	5.3	120	< 200	1.5	0.72

a capsules

TABLE X. - Continued

Specimen (a)	600°C Exposure (hour)	Im						
		Mo	Pt	Na	Nb	Ni	P	Pb
Iridium	1000	< 6	<100	48	270	< 6	< 200	< 20
	2000	1.2	<100	57	670	< 2	< 200	2.1
	3000							No Pota
	5000	0.76	<100	19	290	23	< 200	< 2
Rhodium	1000	< 6	<100	57	1720	< 6	< 200	38
	2000	<0.6	<100	76	210	< 2	< 200	2.1
	3000	<0.6	<100	20	260	5.7	< 200	< 2
	5000	<0.6	<100	23	320	< 6	< 200	< 2
20% Nickel- Clad Silver	1000							No Pota
	2000	13	<200	84	670	190	< 200	11
	3000	<0.6	<100	23	610	15	< 200	48
	5000	1.8	<100	23	610	15	< 200	15
20% Inconel- Clad Silver	1000	< 6	<100	48	<20	< 6	< 200	< 20
	2000	2.9	<100	210	550	150	< 200	9.2
	3000	0.6	<100	23	270	38	< 200	11
	5000	<0.6	<100	19	250	< 6	< 200	2
Blank Standard (Cb-1Zr)	1000	< 6	<100	44	67	< 6	< 200	< 20
	2000	1.9	<100	57	110	9	< 200	< 6
	3000	<0.6	<100	92	710	36	< 200	18
	5000	<0.6	<100	67	110	9.9	< 200	11
Batch Analysis of Potassium		< 3		4		< 5		< 5
Blank Standard (Cb-1Zr) 23°C		< 6	<100	48	42	6	< 200	< 20

(a) Conductors were exposed in columbium-1% zirconium capsules

Impurity (ppm)									Potassium Recovered (grams)
Pb	Sb	Si	Sr	Ti	Y	Rh	Zr	O ₂	
< 20	< 60	25	< 0.6	< 6	< 2	< 10	20		4.92
2.1	< 60	14	< 0.6	< 2	< 2	< 10	19		6.49
No Potassium Was Found In This Capsule									
< 2	< 60	42	< 0.6	2	< 2	< 10	6.9		4.90
38	< 60	92	< 0.6	21	< 2	< 10	23		4.23
2.1	< 60	14	2.1	< 2	< 2	< 10	5.7		5.15
< 2	< 60	12	< 0.6	< 6	< 2	< 10	< 2		5.67
< 2	< 60	38	< 0.6	< 2	< 2	< 10	5.3		5.57
No Potassium Was Found In This Capsule									
11	< 60	53	< 0.6	< 2	< 2	< 10	9.5		5.05
48	< 60	11	< 0.6	< 6	< 2	< 10	< 6		5.04
15	< 60	59	3.8	< 2	86	< 10	12		5.10
< 20	< 60	27	< 0.6	< 6	< 2	< 10	< 20		2.44
9.2	< 60	38	< 0.6	< 2	< 2	< 10	29		5.89
11	< 60	14	< 6	< 6	< 2	< 10	< 6		5.89
2	< 60	40	< 0.6	< 2	< 2	< 10	5.7		5.91
< 20	< 60	6.7	< 0.6	< 6	< 2	< 10	< 20	8.2	3.41
< 6	< 60	46	< 0.6	< 2	< 2	< 10	< 6	69	1.87
18	< 60	210	< 2	< 2	< 2	< 10	7.6	11	1.08
11	< 60	130	< 2	15	< 2	< 10	3.8	7	2.54
< 5		12	< 1	< 5			< 10	< 10	
< 20	< 60	10	< 0.6	< 6	< 2	< 10	< 20	324	2.59

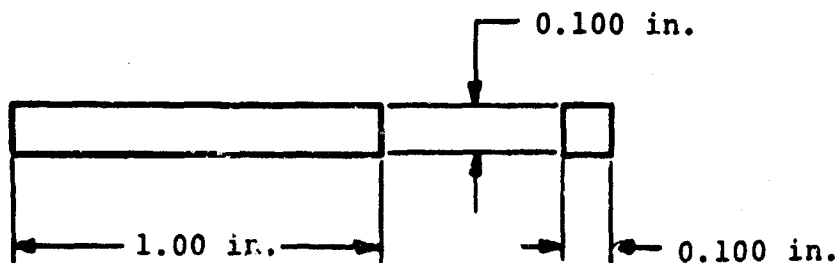
3. Evaluation of Potassium Vapor Exposed Insulations

Insulation specimens were obtained either from commercial suppliers, fabricated at WAED, or a combination of both. Specimens were in the forms of either solid modulus-of-rupture (MOR) bars, shown in figure 18, or flexible felts or wool. Table XI lists materials and suppliers.

a. Cleaning and Preparing Ceramic Specimens

All specimens were prepared for loading by cleaning. Modulus-of-rupture bars stored at WAED were contaminated with a high melting temperature wax used to fixture them during diamond slicing. These bars were prepared by cleaning according to the following schedule:

- 1) Soak in methyl alcohol.
- 2) Scrape off any adhering material-Boil in dilute Alconox detergent solution for 15 minutes.
- 3) Rinse in flowing de-ionized water for 15 minutes.
- 4) Rinse three times in methyl alcohol.
- 5) Dry-vapor degrease in isopropyl alcohol for 15 minutes.
- 6) Dry in an air furnace 100°C for 30 minutes.
- 7) Cool and store in desiccator.



Opposite sides were parallel within 0.001 in.

Tolerances:

Length ± 0.010 in.

Other Dimensions ± 0.001 in.

All surfaces were diamond ground.

Figure 18. Ceramic Modulus-of-Rupture Bar

TABLE XI. Long Term Potassium Vapor Exposure Ceramic Materials

Material	Supplier
Alite A-610 (Bar) ($99\% \text{Al}_2\text{O}_3$ - 1% MgO)	U. S. Stoneware, Akron, Ohio
Strontium Zirconate-(Fused Bar) (SrZrO_3)	Westinghouse Electric Corporation Lima, Ohio, (Fabrication) and Muscle Shoals Electrochemical Corporation, Tennessee (material)
Coors AD999 (Bar) (99.9% Al_2O_3)	Coors Porcelain Company, Golden, Colorado
Alumina-Yttria (Bar) Eutectic Composition (Al_2O_3 - Y_2O_3)	Westinghouse Electric Corporation Lima, Ohio (Fabrication) Linde Division Union Carbide Corporation and Trancoa Company.
Sapphire (felt) (99.99% Al_2O_3)	Thermokinetic Fiber Inc. (materials) Nutley, New Jersey
Sapphire [(felt + $\text{Al}(\text{OCl})_n$)]	Thermokinetic Fibers Inc., Nutley, New Jersey; (felt) and Westinghouse Electric Corporation Lima, Ohio, [$\text{Al}(\text{OCl})_n$]
Beryllium Oxide (Bar) ($99.8\% \text{BeO}$)	Brush Beryllium Co., Elmore, Ohio
Chemically Pure Strontium Zirconate (Bar) (SrZrO_3)	Westinghouse Electric Corporation Lima, Ohio; (Fabrication) and Titanium Alloy Manufacturing Company (material)
Boron Nitride (Wool)	Carborundum Corporation
Yttria (Bar) (Y_2O_3)	Trancoa (material) and Westinghouse Electric Corporation Lima, Ohio (Fabrication)

Purchased MOR bars were cleaned by rinsing in chemically pure acetone, then dried and stored as in 6) and 7) above. Boron nitride has shown resistance to 850°C potassium⁴; therefore, one specimen of felt, made from 99+ $\%$ pure boron nitride fibers by the Carborundum Company, was prepared for 5000-hour 600°C potassium vapor exposure. A 10-minute exposure at 700°C in an air furnace removes any organic material present.

Sapphire mat, made of single crystal whiskers, was prepared by cutting specimens approximately 1/4-inch wide by 2 inches long. Cut specimens were cleared by exposing them to 700°C heat in an air furnace for 10 minutes. One sapphire mat specimen was impregnated with aluminum oxychloride. Impregnation was accomplished by dipping the sapphire mat in a slurry of aluminum oxychloride powder in absolute ethanol. The specimen was then air dried and subsequently heated to 850°C in an air furnace to form alumina from the aluminum oxychloride. This specimen was considerably more rigid than the untreated specimen.

b. Modulus-of-Rupture Test Results

Modulus-of-rupture strengths were determined on unexposed material and after completion of long term exposure. Table XII shows the average MOR values for each group of test specimens before and after exposure to 600°C potassium. The coefficient of variation is also shown to indicate the range of MOR values within each test group. The chemically pure (cp) SrZrO_3 materials' strength declined after 1000 and 3000 hours, decreasing to less than half of its original strength after 5000 hours. SrZrO_3 prepared from fused powder was initially stronger than the cp material. Its strength dropped to about 70% of initial value after 1000 hours and remained at about this level after 2000, 3000, and 5000 hours of exposure. Alite A-610 was exposed for 1000 and 2000 hours only. This material was received as replacement material for Alite A-612 which was inadvertently supplied instead of Alite A-610. Alite A-612 is a 97% Al_2O_3 -3% SiO_2 ceramic that lost all mechanical strength after 1000 hours in 600°C potassium vapor. Alite A-610 lost more than 50% of its strength after 2000 hours. The 1000-hour exposure average strength reported in table XII is less than that reported for the 2000-hour exposure test. The 1000-hour potassium exposure could have been contaminated with a small amount of oxygen and if true, this was a more severe test.

The other materials, Y_2O_3 , BeO Coors AD999, and Al_2O_3/Y_2O_3 , do not show significant changes in strength during any of the exposure time intervals. In some instances the average strength was higher after exposure to potassium than before exposure and some materials show lower strength values at intermediate durations than after 5000 hours. These strength value variations are due to minor variations in material composition or structure.

Figure 19 summarizes these data in bar graph form by comparing strengths before exposure and after exposure for 5000 hours. As discussed previously, both types of $SrZrO_3$ and Alite A-610 show a definite loss of strength and the other materials show essentially no degradation in strength under these test conditions. Coors AD999 and the Al_2O_3/Y_2O_3 eutectic composition materials are seen to be considerably stronger than other materials.

c. Micrograph Comparisons Before and After Tests

Microstructure examinations were made on polished and etched specimens of modulus-of-rupture ceramic materials. Specimens for examination were selected from each material type, both before and after 5000 hours of potassium exposure. With one exception, there were no changes in microstructure. The photomicrograph of cp $SrZrO_3$ material shown in figure 20 after 5000 hours

exposure shows that a large number of grains were pulled from the specimen during polishing. The same material before exposure, which was prepared by identical lapping and polishing methods, has no grain pull-outs. This observation correlates with the strength data. Thus it appears that the bond between grains was largely destroyed by exposure to potassium and the lower strength values are a result of this phenomenon. The cp strontium zirconate had a strong sulfide odor immediately after removal from the hot pressing fixture. The materials used to prepare cp $SrZrO_3$ evidently contained traces of sulfites and/or sulfates that were reduced in the carbon hot pressing dies. These sulfur-containing compounds would migrate to grain boundaries and, as they are not thermodynamically stable in 600°C potassium vapor, the grain boundaries would be subject to potassium attack. This is a possible explanation for the observed attack.

TABLE XII. Average Modulus-of-Rupture Strengths Before and After Exposure to 600°C Potassium Vapor

Material*	SrZrO ₃ (Chemically Pure)		SrZrO ₃ (Fused)		Y ₂ O ₃		BeO	
	Average MOR (psi)	Coefficient of Variation (percent)	Average MOR (psi)	Coefficient of Variation (percent)	Average MOR (psi)	Coefficient of Variation (percent)	Average MOR (psi)	Coefficient of Variation (percent)
Before Exposure	17,412	11.4	22,900	10.0	21,800	--	22,556	--
After 1000 hours	11,626	17.4	23,204	5.1	22,188	10.0	21,610	10.0
After 2000 hours			23,665	3.3			21,458	
After 3000 hours	10,271	6.1	23,505	7.1			20,141	
After 5000 hours	6,803	15.4	22,657	5.4	21,476	17.1	21,333	17.1

*See Table II for a full description of materials.

nd

Al ₂ O ₃	BeO		Coors AD999		Al ₂ O ₃ /Y ₂ O ₃		Alite A-610	
Coeffi- cient of Variation (percent)	Average MOR (psi)	Coeffi- cient of Variation (percent)	Average MOR (psi)	Coeffi- cient of Variation (percent)	Average MOR (psi)	Coeffi- cient of Variation (percent)	Average MOR (psi)	Coeffi- cient of Variation (percent)
--	22,556	10.5	63,125	13.0	62,900	2.5	39,458	2.37
10.0	21,610	5.7	51,275	3.6			3,028	6.9
	21,458	7.0	70,968	2.1			7,396	12.7
	20,141	9.8	58,461	8.5	43,742	14.5		
17.1	21,333	4.7	69,742	5.7	57,429	9.3		

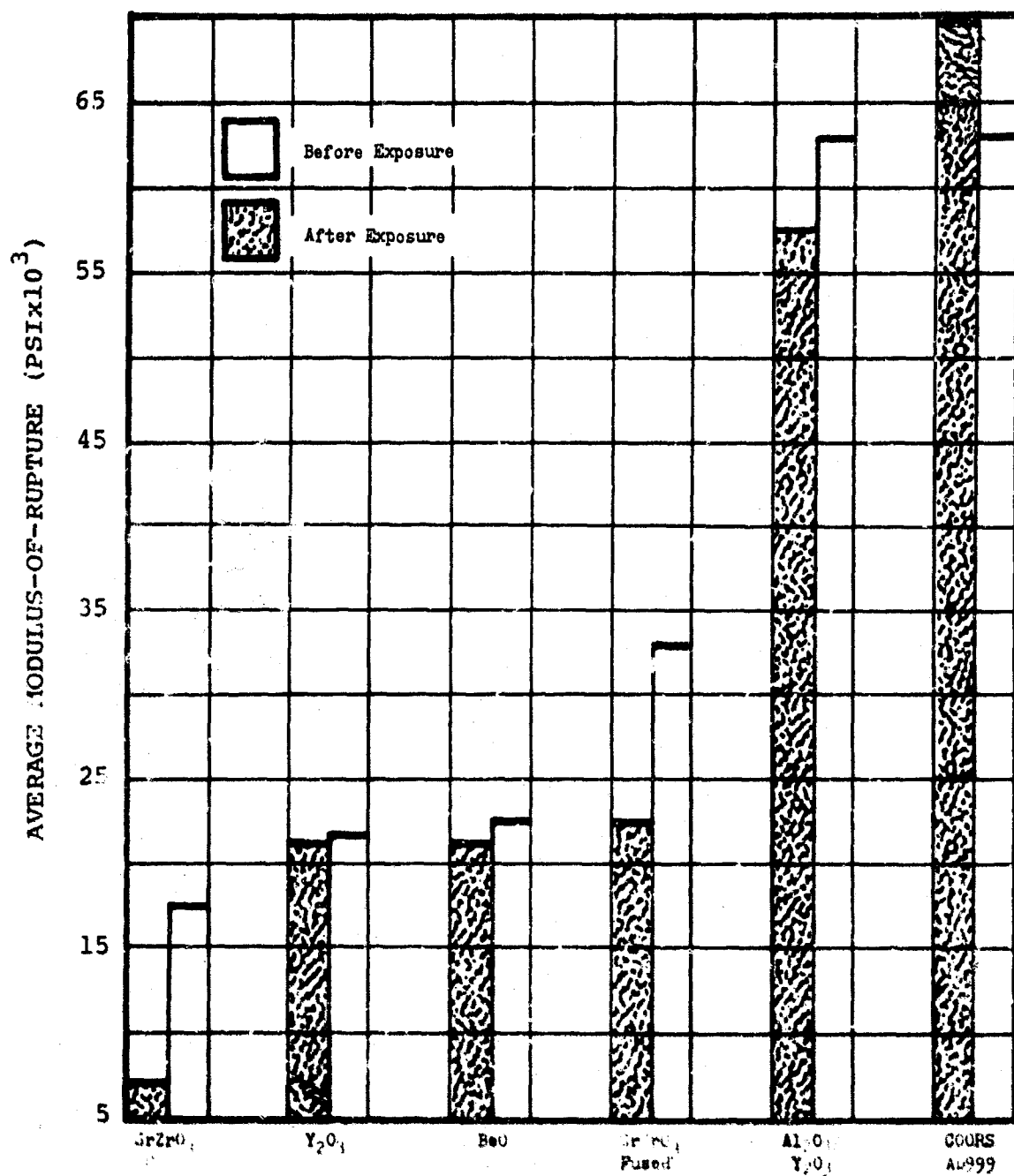
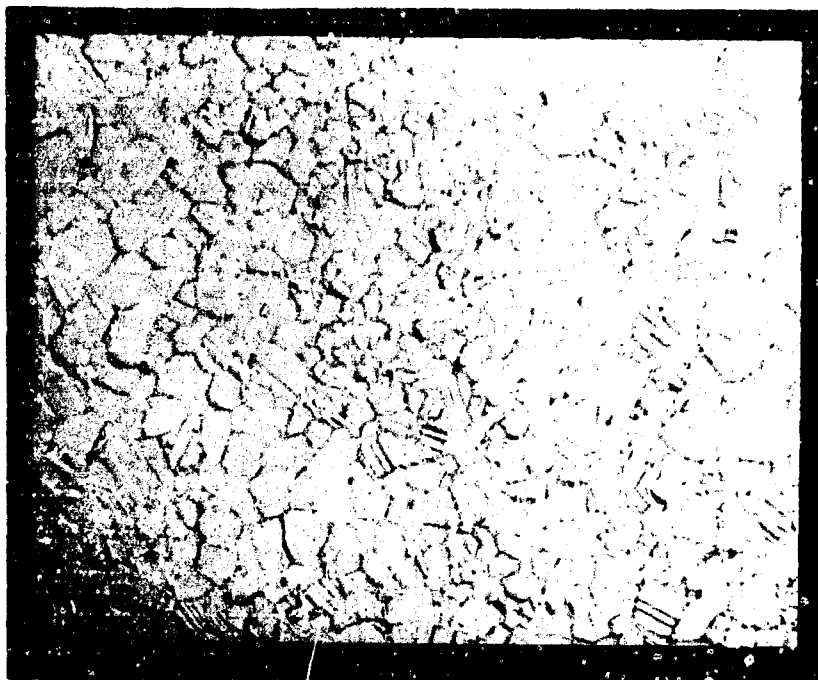


Figure 19. Comparison of Average Modulus-of-Rupture Strength Before and After Exposure to 600°C Potassium for 5000 Hours

CP SrZrO_3
(before exposure
to potassium)

(625X)



CP SrZrO_3
(after exposure
to potassium)

(625X)

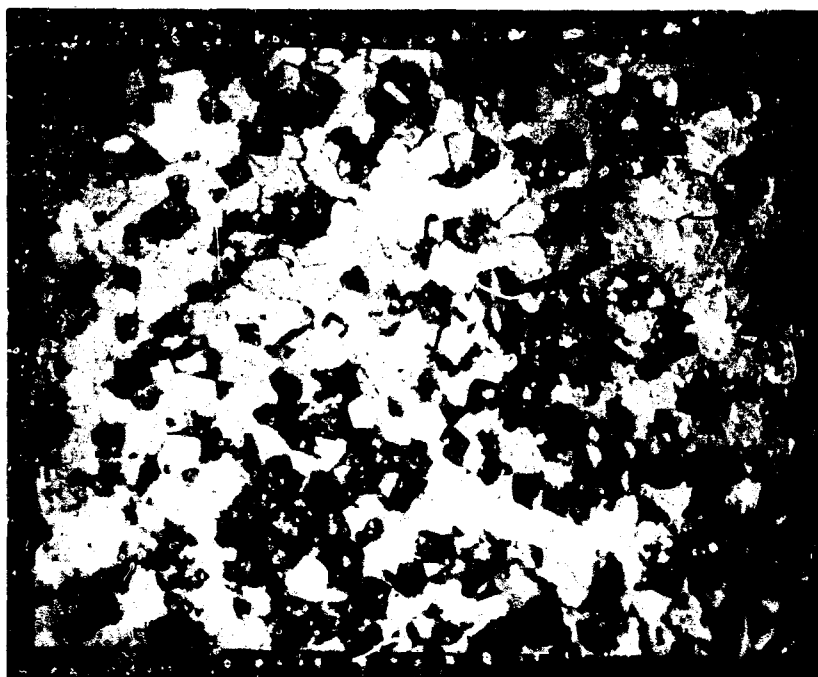


Figure 20. Micrographs of Chemically Pure (cp) Strontium Zirconate Before and After 600°C Potassium Vapor Exposure

Alite A-610 and Alite A-612 are shown in figure 21 before potassium tests only. These materials did not have sufficient strength after exposure to be polished.

All of the other materials retained 70% or more of their original strength after potassium exposure and show no apparent changes in microstructure. Figure 22 shows the microstructure of beryllia before and after 600°C potassium exposure. Micrographs of "hot-pressed" yttrium oxide and alumina-yttria are shown in figures 23 and 24. The small grain size of Coors AD999 is shown in figure 25. Figure 26 shows the microstructure of fused strontium zirconate before and after 600°C potassium exposure. This type of strontium zirconate did not show grain pull-outs due to polishing in contrast to the cp strontium zirconate.

d. Evaluation of Impurity Pickup Analyses

Potassium from ceramic specimen and standard exposure capsules was analyzed to determine the degree of elemental contamination. Table XIII shows the trace element composition of the potassium metal after various exposure times.

Some correlation was observed between elemental pickup by the potassium due to solution or reaction, the MOR values, and microstructure. Figure 19 shows the average MOR strength for ceramic materials before and after exposure to 600°C potassium vapor and indicates loss of strength of the cp strontium zirconate (SrZrO_3)

specimens. Figure 20 shows evidence of attack in the microstructure of this material. The strontium and zirconium impurity levels in the potassium exposed to this material increased appreciably after potassium vapor exposure. Differences in impurity levels are also shown between cp SrZrO_3 and fused SrZrO_3 .

The other ceramic materials exposed to 600°C potassium vapor were not appreciably attacked as shown by the trace element analysis, microstructure examinations, and the MOR tests.

The boron nitride wool was exposed for 5000 hours only. Analysis of the potassium from this exposure showed 330 ppm boron in solution. The boron nitride wool was intact and had a gray surface, caused either by material transport of the container material or reduction to elemental boron. Boron nitride wool shows some promise as an elec-

trical insulating material resistant to 600°C potassium vapor.

Analysis of the potassium from 5000-hour exposed sapphire mat plus Al_2O_3 (formed by decomposing aluminum oxychloride on the mat indicated the combination was not affected by the 600°C potassium vapor. The other sapphire mat specimens (1000-, 2000-, and 3000-hour exposure) did not have aluminum oxychloride decomposed on their surfaces and were also unaffected by exposure tests.

4. Evaluation of Potassium Vapor Exposed Magnetic Materials

Two types of magnetic materials were purchased, processed, given potassium and/or thermal exposure, tested, examined, and evaluated. Both types of materials were unaffected by potassium exposure.

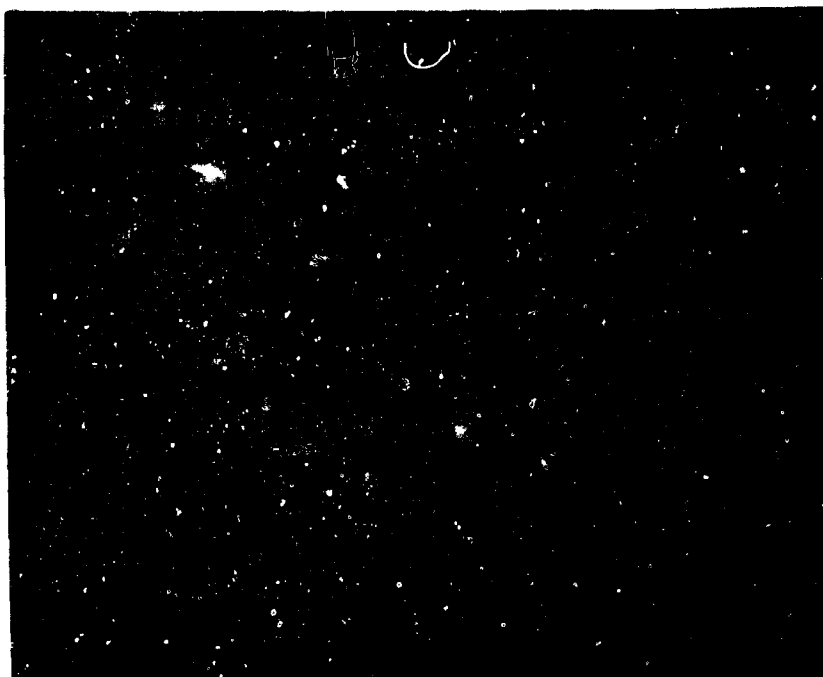
a. Specimen Preparation

Two types of magnetic materials that had previously demonstrated suitability for high temperature potassium vapor applications were evaluated^{5,6}. Table XIV describes Hiperco 27 alloy, gives its chemical composition, and lists its supplier. All Hiperco 27 alloy used on this program was from the same coil. Cubex, a nominal 3.25% silicon iron alloy with double grain orientation, was obtained as 0.011-inch-thick strip from Westinghouse Research and Development Laboratories, Pittsburgh, Pennsylvania.

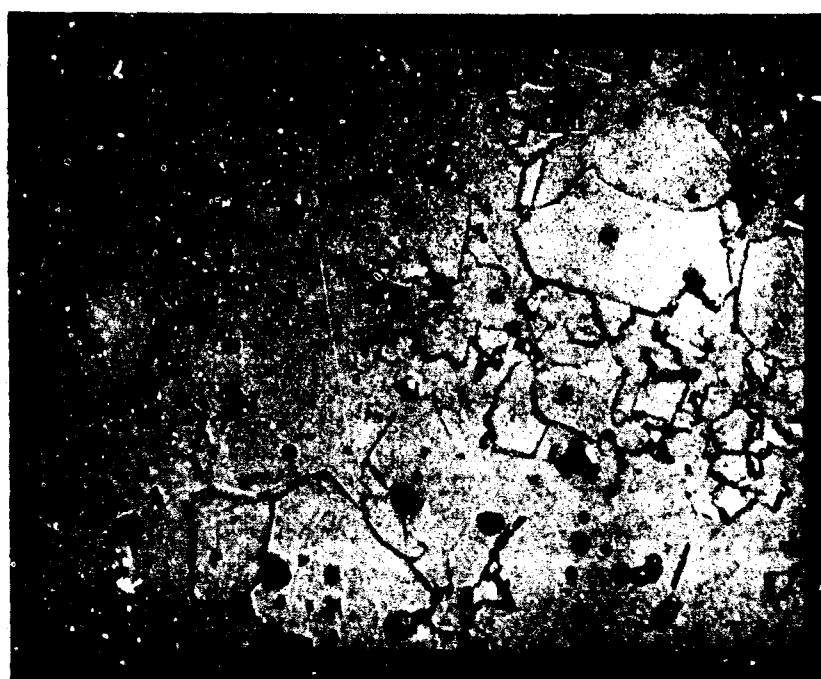
Specimens for mechanical testing were sheared to 0.25-inch wide by 8-inch long size to conform to ASTM test specification A-370. The specimens were cleaned by rinsing in chemically pure acetone.

b. Evaluation of Mechanical Tests

Mechanical tests were performed on magnetic materials. Data from these tests were used to calculate 0.2% offset yield strength and elongation values. Individual values were compared with average values and a calculated standard deviation (see Appendix II). A revised set of values was calculated when a value was outside the standard deviation. Mechanical property values, their averages and the coefficients of variation are given in table XV. Average data from this table are plotted in figure 27. Potassium did not appreciably affect either material.



Alite 612
(before exposure)
(625X)



Alite 610
(before exposure)
(625X)

Figure 21. Micrographs of Alite A-612 and A-610 Before Potassium Vapor Exposure

BeO
(before exposure)
(250X)



BeO
(before exposure)
(250X)

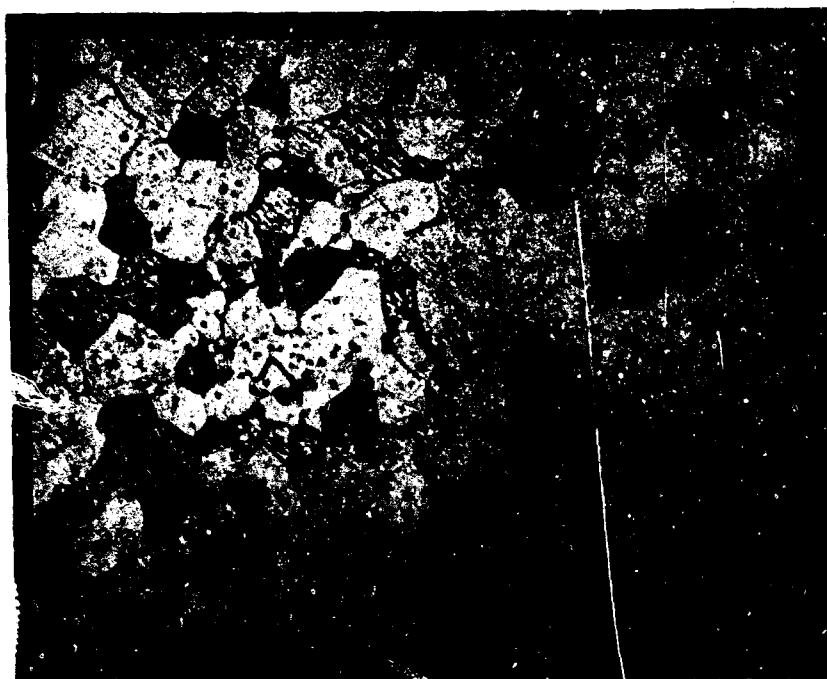
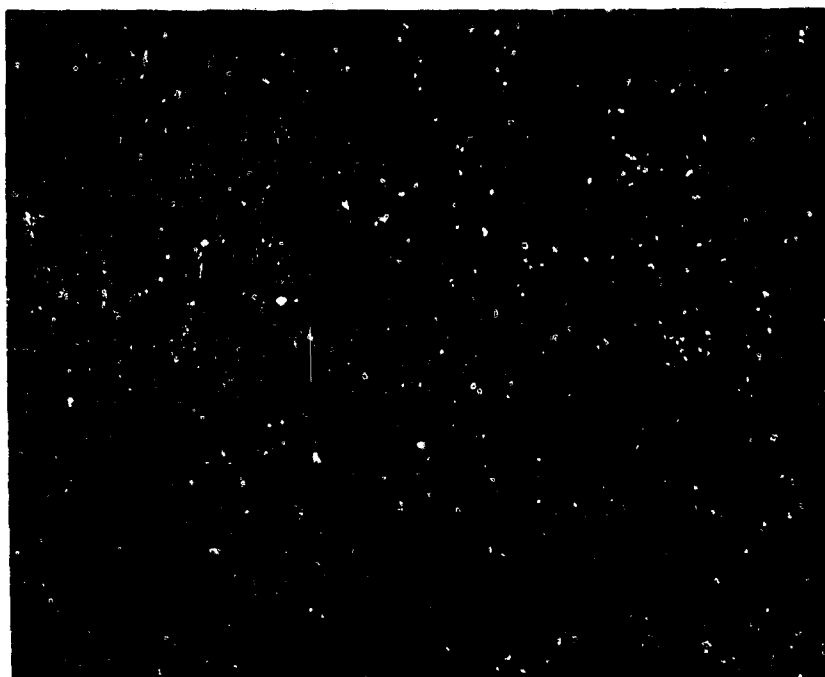


Figure 22. Micrographs of Beryllia Before and After 600°C Potassium Vapor Exposure



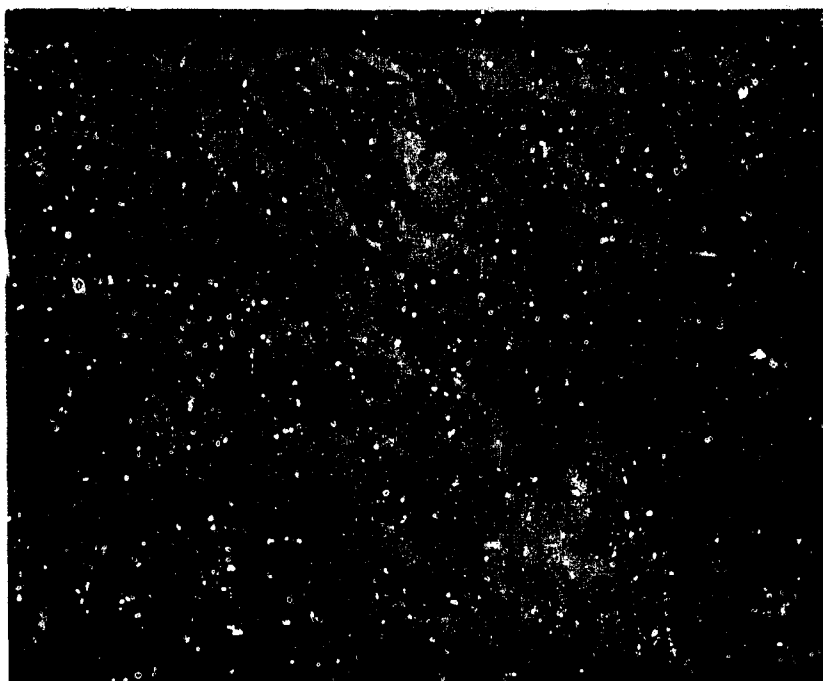
Y_2O_3
(before exposure)
(2500X)



Y_2O_3
(after exposure)
(2500X)

Figure 23. Micrographs of Yttrium Oxide Before and After
600°C Potassium Vapor Exposure

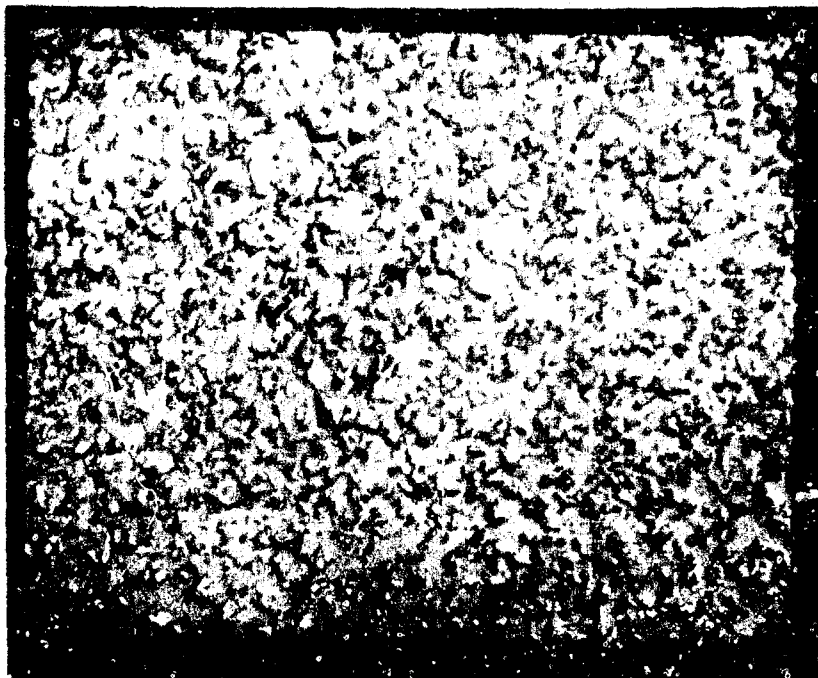
$\text{Al}_2\text{O}_3/\text{Y}_2\text{O}_3$
(before exposure)
(1000X)



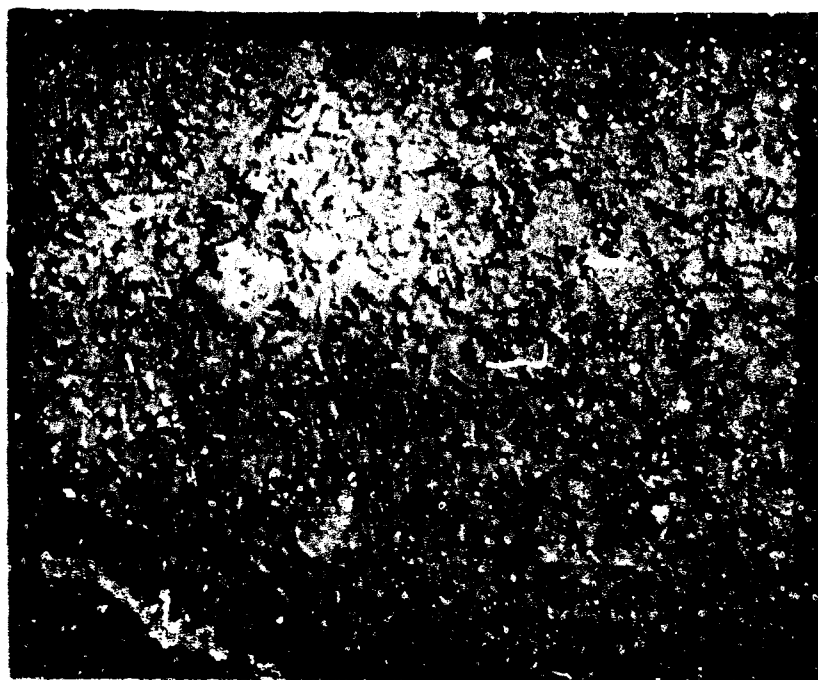
$\text{Al}_2\text{O}_3/\text{Y}_2\text{O}_3$
(after exposure)
(1000X)



Figure 24. Micrographs of Alumina-Yttria Eutectic Before and After 600°C Potassium Vapor



Coors AD999
(before exposure)
(1000X)



Coors AD999
(after exposure)
(1000X)

Figure 25. Micrographs of Coors AD999 Alumina Before and After 600°C Potassium Vapor Exposure

SrZrO_3 (fused)
(before exposure,
(250X)



SrZrO_3 (fused)
(after exposure,
(250X)

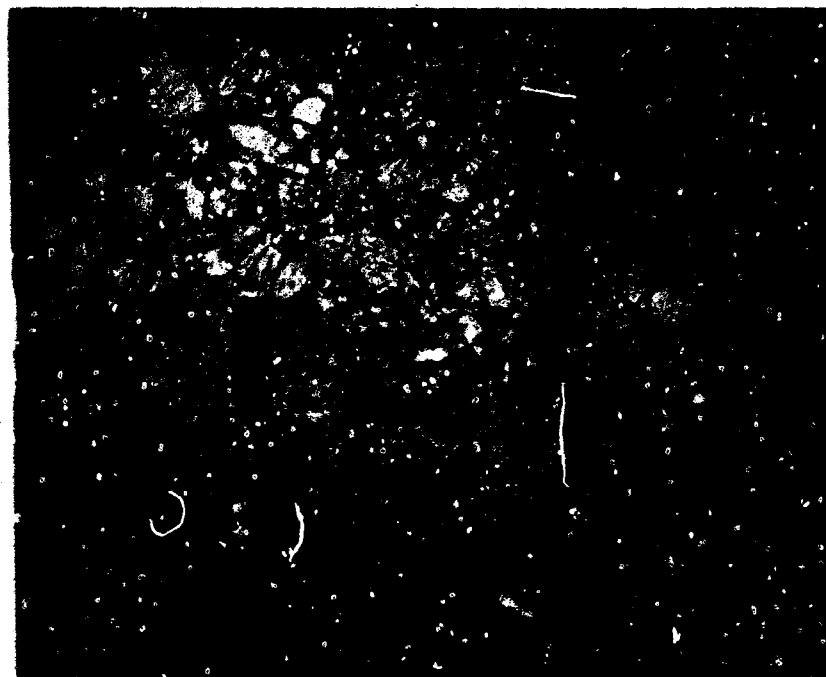


Figure 26. Micrographs of Fused Strontium Zirconate Before
and After 600°C Potassium Vapor Exposure

TABLE XIII. Analyses of Potassium From Ceramic Specimen Exposure Capsule

Specimen (a)	600°C Exposure (hour)							
		Ag	Al	Au	B	Ba	Be	Bi
SrZrO ₃ (Fused)	1000	<0.6	8.0	<600	< 6	< 6	< 2	<20
	2000	< 2	< 6	<100	< 2	< 2	<0.6	< 2
	3000	< 2	34	<100	< 6	< 2	<0.6	< 5
	5000	5.7	< 6	<100	< 6	< 2	<0.6	< 6
	5000	< 2	< 6	<100	< 6	< 2	<0.6	< 6
SrZrO ₃ (C.P.)	1000	20.6	38	600	< 6	< 6	< 2	<20
	2000							No
	3000	< 2	29	<100	< 6	< 2	<0.6	< 6
	5000	< 2	19	<100	< 6	< 2	<0.6	< 6
BeO	1000	<0.6	9.5	<600	< 6	< 6	8.6	<20
	2000	< 2	< 6	<100	< 2	< 2	<0.6	< 2
	3000	< 2	6	<100	< 6	< 2	<0.6	< 6
	5000	4.6	6.0	<100	< 6	< 2	<0.6	< 6
Sapphire Mat	1000	<0.6	19	<600	< 6	< 6	< 2	<20
	2000	< 2	76	<100	< 2	< 2	<0.6	< 2
	3000	2.3	48	<100	< 6	< 2	<0.6	< 6
(Sapphire Mat with Alumina from Aluminum Oxychloride)	5000	3.8	19	<100	< 6	< 2	<0.6	< 6
Y ₂ O ₃	1000	<0.6	7.2	<600	< 6	< 6	< 2	<20
	2000							No
	3000							No
	5000	2.0	19	<100	< 6	< 2	<0.6	< 6

(a) Ceramics were exposed in type 347 Stainless Steel capsules.

Exposure Capsules

Impurity (ppm)											
Ba	Be	Bi	Ca	Cd	Co	Cr	Cu	Fe	Ir	Mg	Mn
6	< 2	<20	< 2	<60	< 6	3.8	17	81	<200	2.1	1.3
2	<0.6	< 2	< 6	< 6	< 2	310	10	990	<100	1.5	51
2	<0.6	< 6	9.5	< 6	< 2	19	31	57	<100	1.9	1.2
2	<0.6	< 6	9.5	< 6	< 2	7.6	6.0	23	<200	<0.6	1.4
2	<0.6	< 6	5.7	< 6	< 2	6.0	7.6	57	<100	<0.6	0.86
6	< 2	<20	< 2	<60	< 6	2.7	6.3	18	<200	0.88	<0.6
No Specimens Exposed											
2	<0.6	< 6	5.7	< 6	< 2	23	29	95	<100	5.3	2.3
2	<0.6	< 6	7.6	< 6	< 6	230	6.0	460	<100	1.5	21
6	8.6	<20	< 2	<60	< 6	120	10	19	<200	1.7	3.8
2	<0.6	< 2	19	< 6	< 2	< 6	6.7	19	<100	50	3.2
2	<0.6	< 6	34	< 6	< 2	< 6	13	23	<100	4.0	0.76
2	<0.6	< 6	9.5	< 6	< 2	8.2	7.6	48	<100	2.5	2.1
6	< 2	<20	5.7	<60	< 6	5.7	5.0	36	<200	1.9	4.2
2	<0.6	< 2	38	< 6	< 2	< 6	8.6	31	<100	5.3	4.2
2	<0.6	< 6	38	< 6	< 2	6.0	15	40	<100	2.9	0.76
2	<0.6	< 6	< 6	6	< 2	<6.0	< 6	40	<100	0.76	2.7
6	< 2	<20	9.5	<60	< 6	7.6	9.5	92	<200	2.5	2.1
No Specimens Exposed											
2	<0.6	< 6	9.5	< 6	< 2	12	9.5	48	<100	6.5	2.5
No Specimens Exposed											

1 capsules.

TABLE XIII. - Continued

Specimen(a)	600°C Exposure (hour)								Imp
		Mo	Pt	Na	Nb	Ni	P	Pb	
SrZrO ₃ (Fused)	1000	< 6	<100	67	<20	6	<200	<20	
	2000	<2.7	<100	48	20	570	<200	< 2	
	3000	0.86	<100	38	140	2.0	<200	<2.0	
	5000	<0.6	<100	20	19	11	<200	< 2	
	5000	<0.6	<100	29	< 6	7.1	<200	< 2	
SrZrO ₃ (C.P.)	1000	< 6	<100	61	<20	< 6	<200	<20	
	2000								No Spe
	3000	0.60	<100	38	19	7.2	<200	3.2	
	5000	2.9	<100	20	19	270	<200	< 2	
BeO	1000	< 6	<100	53	<20	< 6	<200	<20	
	2000	0.6	<100	19	<20	< 2	<200	< 2	
	3000	<0.6	<100	38	<20	< 2	<200	3.4	
	5000	<0.6	<100	19	< 6	< 6	<200	< 2	
Sapphire Mat	1000	< 6	<100	50	<20	6	<200	<20	
	2000	<0.6	<100	72	<20	< 2	<200	< 2	
	3000	<0.6	<100	19	<20	38	<200	< 2	
(Sapphire Mat with Alumina from Aluminum Oxychloride)	5000	0.6	<100	29	12	6.0	<200	< 2	
Y ₂ O ₃	1000	< 6	<100	63	<20	6	<200	<20	
	2000								No Spe
	3000								No Spe
	5000	0.60	<100	38	14	6.0	<200	< 2	

(a) Ceramics were exposed in type 347 Stainless Steel capsules.

Impurity (ppm)										Potassium Recovered (grams)
P	Pb	Sb	Si	Sr	Ti	Y	Rh	Zr	O ₂	
<200	<20	<60	42	48	9.5	< 2	<10	38		1.91
<200	< 2	<60	48	19	< 2	< 2	<10	11		5.25
<200	<2.0	<60	23	3.6	< 2	< 2	<10	< 6		5.31
<200	< 2	<60	13	5.3	< 6	< 2	<10	< 2		4.62
<200	< 2	<60	14	5.3	< 6	< 2	<10	< 2		4.67
<200	<20	<60	46	9.5	< 6	< 2	<10	<20		3.86
No Specimens Exposed										
<200	3.2	<60	21	44	< 2	< 2	<10	76		5.24
<200	< 2	<60	55	42	< 6	< 2	<10	2.3		5.38
<200	<20	<60	59	<0.6	13	< 2	<10	<20		3.05
<200	< 2	<60	40	<0.6	2	< 2	<10	< 2		4.88
<200	3.4	<60	27	<0.6	6	< 2	<10	< 6		2.60
<200	< 2	<60	31	<0.6	<6.0	< 2	<10	< 2		4.52
<200	<20	<60	12	<0.6	12	< 2	<10	<20		3.87
<200	< 2	<60	42	<0.6	2	< 2	<10	< 2		4.14
<200	< 2	<60	<0.6	<0.6	< 6	< 2	<10	< 6		4.62
<200	< 2	<60	27	<0.6	< 6	< 2	<10	< 6		5.16
<200	<20	<60	18	<0.6	6.7	130	<10	<20		2.19
No Specimens Exposed										
No Specimens Exposed										
<200	< 2	<60	74	2.3	2.3	86	<10	< 2		4.77

ules.

TABLE XIII. - Continued

Specimen (a)	600°C Exposure (hour)							
		Ag	Al	Au	B	Ba	Be	
Coors AD999	1000	<0.6	11	<600	< 6	< 6	< 2	<
	2000	< 2	6.0	<100	< 2	< 2	<0.6	<
	3000	2	< 6	<100	< 6	< 2	<0.6	<
	5000	3.8	< 6	<100	< 6	< 2	<0.6	<
Alumina-Yttria Eutectic	1000							
	2000							
	3000	< 2	19	<100	< 6	< 2	<0.5	<
	5000	2.0	15	<100	< 6	< 2	<0.6	<
Blank Standard (347 Stainless Steel) 600°C	1000	<0.6	3.1	<600	< 6	< 6	< 2	<
	2000	< 2	290	<100	<20	<20	< 2	<
	3000	< 2	40	<100	<20	<20	< 2	<
	5000	< 2	480	<100	< 6	< 2	<0.6	<
Blank Standard (Cb-1Zr) 600°C	1000	<0.6	1.9	<600	< 6	< 6	< 2	<
	2000	< 2	9.9	<100	< 6	<20	< 2	<
	3000	2	48	<100	< 6	< 2	< 2	<
	5000	< 2	32	<100	< 6	< 2	0.6	<
Blank Standard (347 Stainless Steel) 23°C		<0.6	6.7	<600	< 6	< 6	< 2	<

(a) Ceramics were exposed in type 347 Stainless Steel capsules.

Impurity (ppm)

Ba	Be	Bi	Ca	Cd	Co	Cr	Cu	Fe	Ir	Mg	Mn
< 6	< 2	<20	5.7	<60	< 6	1.9	5.3	15	<200	0.82	1.8
< 2	<0.6	< 2	21	< 6	< 2	17	5.7	72	<100	2.5	5.9
< 2	<0.6	< 6	19	< 6	< 2	44	11	210	<100	2.3	5.5
< 2	<0.6	< 6	< 6	9.5	< 2	< 6	9.5	19	<100	2.5	1.6
No Specimens Exposed											
< 2	<0.6	< 6	29	< 6	< 2	13	5.7	31	<100	2.5	0.93
< 2	<0.6	< 6	9.5	< 6	< 2	340	7.6	1900	<100	2.5	40
< 6	< 2	<20	7.6	<60	< 6	5.7	14	29	<200	1.3	1.4
<20	< 2	< 6	3.8	<20	< 6	82	21	210	<100	<0.6	8.6
<20	< 2	< 6	5.7	<20	< 6	67	11	110	<100	<0.6	2.1
< 2	<0.6	< 2	< 2	< 6	< 2	130	25	630	<100	2.9	21
< 6	< 2	<20	5.7	<60	< 6	4.8	5.0	170	<200	0.95	1.5
<20	< 2	< 6	3.8	<20	< 6	9.5	23	34	<100	<0.6	1.2
< 2	< 2	< 6	2	<20	< 6	4.8	290	270	<100	7.6	10
< 2	0.6	< 2	< 2	< 6	< 2	1.9	16	23	<100	<0.6	<0.6
< 6	< 2	<20	2.9	<60	< 6	1.9	0.76	5.9	<200	0.59	2.3

capsules.

TABLE XIII. - Concluded

Specimen (a)	600°C Exposure (hour)	Imp						
		Mo	Pt	Na	Nb	Ni	P	Pb
Coors AD999	1000	< 6	<100	59	<20	< 6	<200	<20
	2000	<0.6	<100	38	40	10	<200	< 2
	3000	0.6	<100	19	57	38	<200	3.8
	5000	<0.6	<100	19	13	< 6	<200	< 2
Alumina-Yttria Eutectic	1000							No Sp
	2000							No Sp
	3000	<0.6	100	950	57	5.0	200	2
	5000	4.2	100	34	12	170	200	2
Blank Standard (347 Stainless Steel) 600°C	1000	< 6	100	63	20	7.6	200	20
	2000	3.8	100	63	60	36	200	6
	3000	1.9	100	76	60	15	200	6
	5000	<0.6	100	86	20	130	200	34
Blank Standard (Cb-1Zr) 600°C	1000	< 6	100	44	67	6	200	20
	2000	1.9	100	57	110	9.0	200	6
	3000	<0.6	100	92	7.0	36	200	18
	5000	<0.6	100	67	110	9.9	200	11
Blank Standard (347 Stainless Steel) 23°C		< 6	<100	59	<20	< 6	<200	<20

(a) Ceramics were exposed in type 347 Stainless Steel capsules.

Impurity (ppm)

Potassium
Recovered
(grams)

P	Pb	Sb	Si	Sr	Ti	Y	Rh	Zr	O ₂	
<200	<20	<60	10	<0.6	< 6	< 2	<10	<20		4.60
<200	< 2	<60	38	< 6	<0.6	< 2	<10	< 2		4.65
<200	3.8	<60	23	< 6	<0.6	< 2	< 6	< 2		5.31
<200	< 2	<60	38	< 6	<0.6	< 2	<10	< 2		4.64
No Specimens Exposed										
No Specimens Exposed										
200	2	60	32	5.0	6	2	10	6		4.61
200	2	60	42	0.6	5	2	10	2		5.17
200	20	60	11	0.6	6	2	10	20	10.8	2.87
200	6	60	57	0.6	2	2	10	6	9.0	1.91
200	6	60	54	0.6	2	2	10	6	15	2.01
200	34	60	170	2	2.9	2	10	4.8	21	1.93
200	20	60	6.7	0.6	6	2	10	20	8.2	3.41
200	6	60	46	0.6	2	2	10	6	69	1.87
200	18	60	210	2	2	2	10	7.6	11	1.08
200	11	60	130	2	15	2	10	3.8	7	2.54
<200	<20	<60	15	0.95	< 6	< 2	<10	<20	11.5	2.88

es.

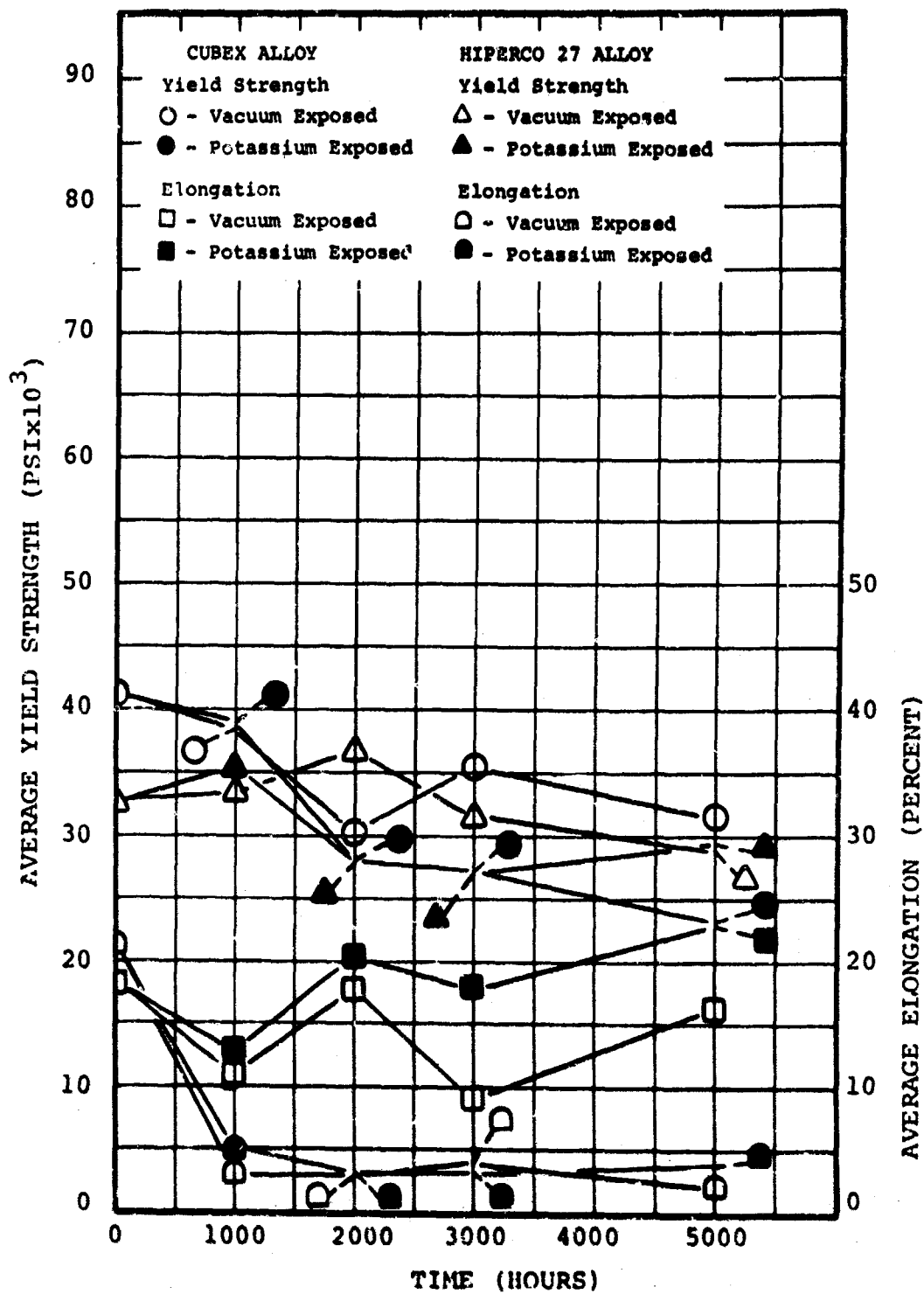


Figure 27. Average Mechanical Test Data for Magnetic Materials

TABLE XIV. Hiperco 27 Alloy Chemical Composition

Heat No. HO 8084		
<u>Element</u>	<u>Specification (%)</u>	<u>Analysis (%)</u>
N	0.70 max.	0.08
S	0.025 max.	0.0056
Si	0.25 max.	0.17
Mn	0.50 max.	0.33
C	0.020 max.	0.020
Co	26.00-28.50	27.8
Cu	-	< 0.10
P	0.015 max.	0.003
Cr	0.30-0.70	0.59
Fe	Balance	-
Material Description: Strip 0.008-inch thick, 14 inches wide.		
Material Supplier: Westinghouse Materials Manufacturing Division, Blairsville, Pa.		

TABLE XV. Mechanical Properties of Unexposed and Long Term Potassium Exposed Magnetic Materials

Material	Unexposed			1000 Hour Exposed			2000 Hour Exposed	
	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)
Hiperco 27 Alloy								
Vacuum	33183 32432 33183	32933	1.07	34756 33203 32945	33635	2.38	36764 38300 37100 35500	
Potassium				35960 34975 35644	35526	1.15	30392 27450 26237	
Material	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)
Hiperco 27 Alloy								
Vacuum	21 22 22.5	21.83	2.862	3 3 3	3	0	3 3 3 3	
Potassium				6 4	5	20	3 3.5 3 3	

Material	Unexposed			1000 Hour Exposed			2000 Hour Exposed	
	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)
Cubex Alloy								
Vacuum	41857 41218 41481	41517	6.25	38433 39403 29048	38961	1.03	29300 35600 27300	
Potassium				38418 40404 38503	39108	2.34	28407 27371 29770	
Material	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)	Coefficient of Variation (percent)	Elongation in 2 inches (percent)	Average Elongation in 2 inches (percent)
Cubex Alloy								
Vacuum	22.5 15 17.5	18.3	17.0	12.5 10	11.25	11.11	18 18 18	
Potassium				14 12.5	13.25	5.66	19 23 20	

ng Term

Client Location	2000 Hour Exposed			3000 Hour Exposed			5000 Hour Exposed		
	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)
04	36764 38300 37100 35500	36913	2.1	30300 32700 32300	31767	3.30	31372 28571 27227	29057	5.9
	30392 27450 26237	28026	6.22	27450 24357 29411	27204	7.02	29702 29556 24702	29653	0.23
Client Location	Elonga- tion in 2 inches (percent)	Average Elonga- tion in 2 inches (percent)	Coefficient of Variation (percent)	Elonga- tion in 2 inches (percent)	Average Elonga- tion in 2 inches (percent)	Coefficient of Variation (percent)	Elonga- tion in 2 inches (percent)	Average Elonga- tion in 2 inches (percent)	Coefficient of Variation (percent)
	3 3 3 3	3	9	4 5 4 3	4	17.67	3 3 2 2	2.5	20
	3 3.5 3 3	3.125	6.9	4 3 4 3	3.5	14.3	3.5 4 3.5 3.5	3.6	6.0

Client Location	2000 Hour Exposed			3000 Hour Exposed			5000 Hour Exposed		
	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)	Yield Strength (psi)	Average Yield Strength (psi)	Coefficient of Variation (percent)
03	29300 35600 27300	30400	12.75	36700 34900 35900	35833	7.05	33207 32452 29811	31823	4.58
	28407 27371 29770	28516	3.44	28464 27203 28044	27904	1.88	27205 23306 21033	23880	10.84
Client Location	Elonga- tion in 2 inches (percent)	Average Elonga- tion in 2 inches (percent)	Coefficient of Variation (percent)	Elonga- tion in 2 inches (percent)	Average Elonga- tion in 2 inches (percent)	Coefficient of Variation (percent)	Elonga- tion in 2 inches (percent)	Average Elonga- tion in 2 inches (percent)	Coefficient of Variation (percent)
	18 18 8	18	0	9 10 9	9.3	5.0	15	16.5	9.1
66	4 23 20	20.7	8.2	16 20 19 18	18.25	8.1	22 25 23 23	23.25	4.7

c. Evaluation of Microstructures

Specimens of magnetic materials were obtained after each exposure duration and prior to mechanical testing. These specimens were mounted, polished, and etched, and given microstructure examinations both before and after etching. Specimens were etched as shown in table XVI. Since there was no apparent change over the period of test only the 5000-hour photomicrographs are included. Figure 28 shows vacuum and potassium vapor exposed Cubex alloy after 5000 hours at 600°C. No significant difference as a result of potassium exposure was seen. The potassium vapor exposed sample shows some etch pitting and a deep grain boundary etch. The vacuum sample also shows some staining.

Figure 29 shows Hiperco 27 alloy after exposure to vacuum and potassium vapor for 5000 hours at 600°C. There was no apparent effect on the microstructure.

d. Evaluation of Impurity Pickup Analyses

Potassium from standard and magnetic material exposure capsules were analyzed for impurities. Table XVII gives these impurity analyses.

The contact of dissimilar metals in the presence of saturated potassium vapor seemed to act as an electrochemical junction accelerating the corrosion or solution of the metals in 600°C potassium. Once the metal is in solution it may be transported to other sites in the

TABLE XVI. Etchants Used on Magnetic Materials

Material	Etchant
Hiperco 27 Alloy	6 Parts HNO_3 4 Parts H_3CCOOH
Cubex Alloy	40% HCl



Thermal Vacuum Exposure

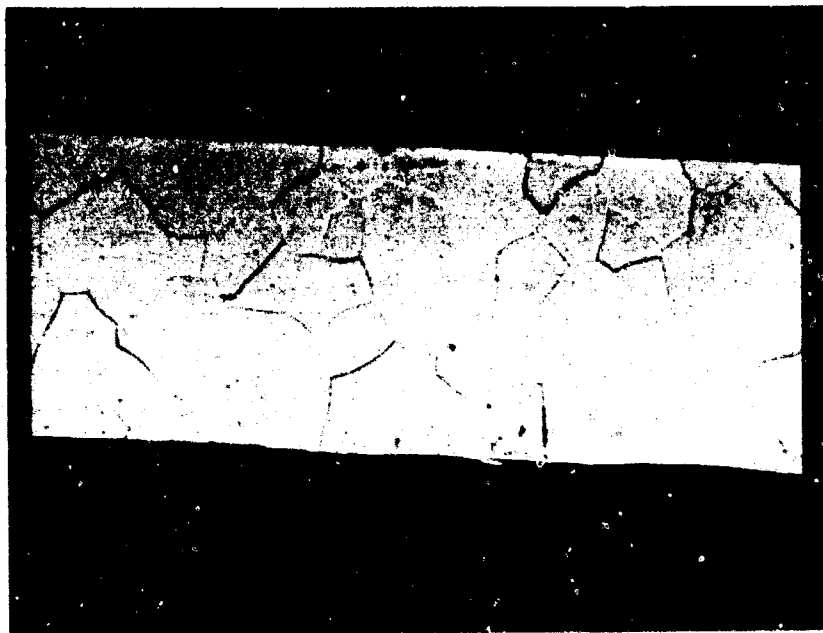


Potassium Vapor Exposure

Figure 28. Micrographs of Cubex Alloy After 5000 Hours at 600°C
Thermal Exposure With and Without Potassium Vapor
(100X)



Thermal Vacuum Exposure



Potassium Vapor Exposure

Figure 29. Micrographs of Hiperco 27 Alloy After 5000 Hours at 600°C Thermal Exposure With and Without Potassium Vapor (200X)

exposure capsule or test specimen. An example of this was seen by observing the increase in the columbium content of the potassium when Hiperco 27 alloy and Cubex alloy were exposed in a columbium-1% zirconium capsule. The columbium level appeared to decrease in the potassium of the Hiperco 27 alloy test capsule after rising to a maximum. The standard at 600°C also rose to a maximum value of 710-ppm Cb and then fell to 110-ppm Cb. An over all survey of the elemental solution from the magnetic materials in 600°C potassium vapor shows there was not appreciable solution of specimen material. No increase in cobalt was found in the potassium from the Hiperco 27 alloy capsule. The potassium from the Cubex alloy capsule was less affected and had less total corrosion products than the potassium from the Hiperco 27 alloy capsule.

5. Systems for Continuously Monitoring Material Performance in 600°C Potassium Vapor

Test systems were designed, fabricated, and used to continuously monitor electrical conductor and magnetic material performance while in potassium vapor. These systems were unique in that only the materials under test, not the activating source, were in potassium vapor. Tests which could be performed were limited by potassium which coated container ceramic parts.

a. Conductor Test Systems

A system to continuously monitor the resistivity of electrical conductors in potassium vapor was designed, fabricated, tested and evaluated. Nickel-clad silver, the transformer conductor, was tested.

(1) Description of Test Containers

(a) General Description

Test containers were made of potassium resistant materials and contained electrical feed-through terminals. Two test containers were fabricated and used in conductor testing. Each container consisted of a 6-inch length of 6-inch diameter schedule 40 pipe in type 347 stainless steel, top and bottom plates of 0.125-inch-thick type 316 stainless steel, a type 347 stainless steel potassium loading and evacuation tube, and a type 304 stainless

TABLE XVII. Analyses of Potassium From Magnetic Material Specimen Exposure

Specimen (a)	600°C Exposure (hour)							
		Ag	Al	Au	B	Ba	Be	Bi
Hiperco 27 Alloy	1000	<0.6	12	<600	< 6	< 6	< 2	<20
	2000	5.9	76	<100	3.8	3.8	<0.6	< 2
	3000 (b)	140	570	<1000	95	<20	< 6	<60
	5000	< 2	23	<100	< 6	< 2	<0.6	< 6
Cubex Alloy	1000	<0.6	12	<600	< 6	< 6	< 2	<20
	2000	< 2	9.5	<100	< 2	< 2	<0.6	< 2
	3000	< 2	< 6	<100	< 6	< 2	<0.6	< 6
	5000	< 2	29	<100	< 6	< 2	<0.6	< 6
Blank Standard (Cb-1Zr)	1000	<0.6	1.9	<600	< 6	< 6	< 2	<20
	2000	< 2	9.9	<100	< 6	<20	< 2	< 6
	3000	2	48	<100	< 6	< 2	< 2	< 6
	5000	< 2	32	<100	< 6	< 2	<0.6	< 2
Potassium Supplier's Batch Analysis		< 1	< 2		<10	< 3	< 1	
Blank Standard (Cb-1Zr) 23°C		<0.6	4.8	<600	< 6	< 6	< 2	<20
Blank Standard (347 Stainless Steel) 23°C		<0.6	6.7	<600	< 6	< 6	< 2	<20
(a) Hiperco 27 Alloy and Cubex were exposed in columbium-1% zirconium								
(b) Only 0.01 gram of potassium was recovered from this capsule; there								

Specimen Exposure Capsules

Impurity (ppm)

	Be	Bi	Ca	Cd	Co	Cr	Cu	Fe	Ir	Mg	Mn
	< 2	<20	< 2	<60	< 6	0.57	4.4	5.7	<200	3.8	<0.6
	<0.6	< 2	38	60	< 2	10	27	86	<100	10	31
	< 6	<60	430	<60	59	84	190	3430	<1000	570	4.6
	<0.6	< 6	15	< 6	< 2	< 6	67	27	<100	4.6	1.1
	< 2	<20	2	<60	< 6	0.95	5	51	<200	2.7	<0.6
	<0.6	< 2	23	<60	< 2	< 6	14	46	<100	6.7	0.57
	<0.6	< 6	< 6	<60	< 2	6.0	5.7	15	<100	1.3	0.57
	<0.6	< 6	< 6	< 6	< 2	< 6	19	27	<100	6.1	<0.6
	< 2	<20	5.7	<60	< 6	4.8	5.8	170	<200	0.95	1.5
	< 2	< 6	3.8	<20	< 6	9.5	23	34	<100	<0.6	1.2
	< 2	< 6	2	<20	< 6	48	290	270	<100	7.6	10
	<0.6	< 2	< 2	< 6	< 2	1.9	16	23	<100	<0.6	<0.6
	< 1		1		< 5	< 5	2	5		< 2	< 1
	< 2	<20	5.7	<60	< 6	4.8	5.3	120	<200	1.5	0.72
	< 2	<20	2.9	<60	< 6	1.9	0.76	5.9	<200	0.59	2.3

m-1% zirconium capsules.

capsule; therefore, impurity analyses are not significant.

TABLE XVII. - Continued

Specimen(a)	600°C Exposure (hour)							
		Mo	Pt	Na	Nb	Ni	P	Pb
Hiperco 27 Alloy	1000	< 6	<100	44	110	< 6	<200	<20
	2000	1.4	<200	100	4770	6.1	<200	3.8
	3000(b)	21	<100	2100	5700	92	<2000	50
	5000	1.7	<100	67	270	< 6	<200	< 2
Cubex Alloy	1000	< 6	<100	50	400	< 6	<200	<20
	2000	<0.6	<200	74	100	1.9	<200	1.9
	3000	<0.6	<200	19	110	3.8	<200	< 2
	5000	0.6	<100	29	1080	< 6	<200	< 2
Blank Standard (Cb-1Zr)	1000	< 6	<100	44	67	< 6	<200	<20
	2000	1.9	<100	57	110	9	<200	< 6
	3000	<0.6	<100	92	710	36	<200	18
	5000	<0.6	<100	67	110	9.9	<200	11
Potassium Supplier's Batch Analysis		< 3		4		< 5		5
Blank Standard (Cb-1Zr) 23°C		< 6	<100	48	42	6	<200	<20
Blank Standard (347 Stainless Steel) 23°C		< 6	<100	59	<20	< 6	<200	<20

(a) Hiperco 27 Alloy and Cubex were exposed in columbium-1% zirconium c

(b) Only 0.01 gram of potassium was recovered from this capsule; theref

Impurity (ppm)											Potassium Recovered (grams)
Ni	P	Pb	Sb	Si	Sr	Ti	Y	Rh	Zr	O ₂	
< 6	<200	<20	<60	44	<0.6	< 6	< 2	<10	<20		4.37
6.1	<200	3.8	<60	61	< 6	< 2	38	<10	53		2.00
92	<2000	50	<600	360	190	<60	<20	<100	150		0.01
< 6	<200	< 2	<60	42	3.8	2.9	86	<10	19		5.22
< 6	<200	<20	<60	59	<0.6	< 6	< 2	<10	<20		5.81
1.9	<200	1.9	<60	51	<0.6	2	< 2	<10	71		2.33
3.8	<200	< 2	<60	19	2.9	< 6	< 2	<10	< 6		5.71
< 6	<200	< 2	<60	48	<0.6	2.3	86	<10	8.8		5.28
< 6	<200	<20	<60	6.7	<0.6	< 6	< 2	<10	<20	8.2	3.41
9	<200	< 6	<60	46	<0.6	< 2	< 2	<10	< 6	69	1.87
36	<200	18	<60	210	< 2	< 2	< 2	<10	7.6	11	1.08
9.9	<200	11	<60	130	< 2	15	< 2	<10	3.8	7	2.54
< 5		5		12	< 1	< 5			<10	<10	
6	<200	<20	<60	10	<0.6	< 6	< 2	<10	<20	32.4	2.59
< 6	<200	<20	<60	15	0.95	< 6	< 2	<10	<20	11.5	2.88

Alum-1% zirconium capsules.

is capsule; therefore, impurity analyses are not significant.

steel thermocouple well. Two electrical feedthrough terminals were used in assembling the first test container and four feedthrough terminals in the second.

(b) Two-Terminal Test Container

Electrical feedthrough terminals for the first container were made at Westinghouse Aerospace Electrical Division. Figure 30 shows the complete top of this container. The feedthrough ceramic members were Lucalox tubes 0.75-inch outside diameter by 0.5-inch inside diameter. One ceramic piece was 1-1/2 inches high and the other two pieces (back-up rings) were 1/8-inch high. These seals were assembled by placing sections of 1/2-inch outside diameter columbium-1% zirconium tube approximately 3/8-inch inside the Lucalox tube, placing formed columbium-1% zirconium washers adjacent to the Lucalox tube, placing the back-up rings adjacent to the washers, and fixturing the assembly by placing columbium-1% zirconium wires in grooves in the tubes.

Braze material was then added to the washers (which had been formed to serve as a reservoir). This braze material was made of the basic alloy described in table XVIII to which 2% copper in the form of unalloyed powder was added. Brazing was accomplished in a cold-wall vacuum furnace

TABLE XVIII. Basic Active Metal Braze Alloy Composition

Element	Analyzed Composition (weight percent)	Remarks
O	0.079	Gas Analysis
N	0.048	Kjeldahl
H	0.0032	Gas Analysis
Ti	48.8	X-ray Fluor.
Cb	19.9	X-ray Fluor.
Ni	32.5	Wet
Fe	est. 0.1	

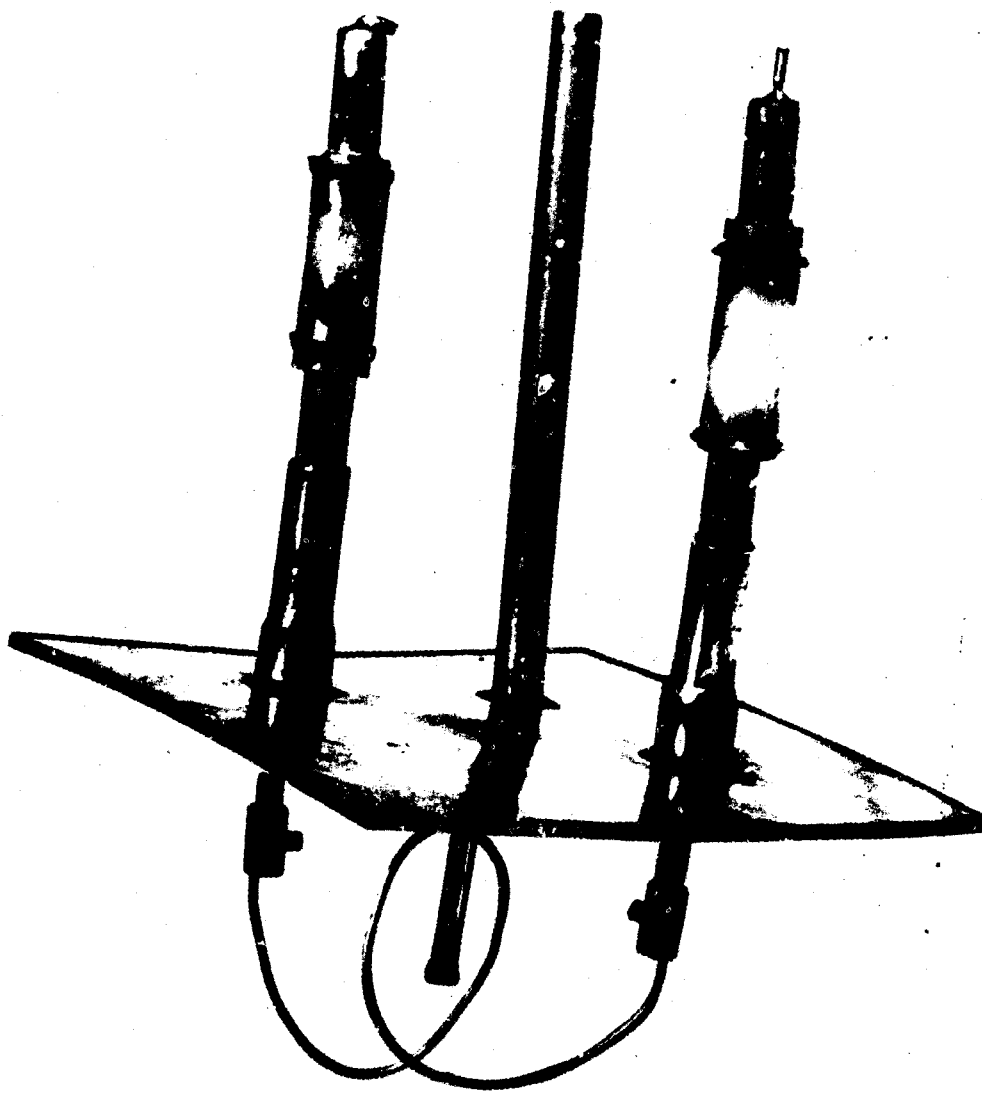


Figure 30. Lid of Two-Terminal Conductor Test Container

(at a pressure of 10^{-6} torr) by radio frequency (rf) heating using a tantalum susceptor. Parts were slowly heated to 1250°C (the slow cycle allowed parts to evolve gas), held at this temperature for five minutes, then cooled as rf power was turned off.

Electrical conductors were added to the feedthroughs by TIG welding 1/8-inch diameter columbium-1% zirconium rods to 1/2-inch diameter washers of the same material and then TIG welding this assembly to one of the metal tubes in the brazed assembly. Welding was done in an inert atmosphere glove box with oxygen and moisture both below one ppm. Feedthrough terminals were completed by brazing (using Microbraz 130) columbium-1% zirconium tubes to 316 stainless steel tubes in a manner similar to that used in the ceramic-to-metal brazing. Helium leak tight integrity was checked after each braze and weld step.

The two-terminal test container top was assembled as shown in figure 30 by TIG welding the stainless steel parts of the feedthrough terminals, the fill and evacuation tube, and the thermocouple well; all to the top plate. Terminal seals were tested for leaks with helium after the top was assembled and twice heated to 700°C . Seals remained helium leak-tight as determined on a CVC helium leak detector (sensitivity limit $<5 \times 10^{-11}$ atm cc/sec).

(c) Four-Terminal Test Container

Feedthrough terminals for the second test container were purchased from EIMAC Division of Varian Associates, and are the same type used in the transformer and window-terminal feedthrough test containers (discussed in Section III.C.6, page 173). Figure 31 shows the construction of these feedthroughs. Four feedthroughs, a potassium loading and evacuation tube, and a thermocouple well were TIG welded into the top plate of the container. Figure 32 shows the completed top and the bottom portion of this test container.

Terminal feedthroughs were tested for leaks with helium before assembly into the top plate.

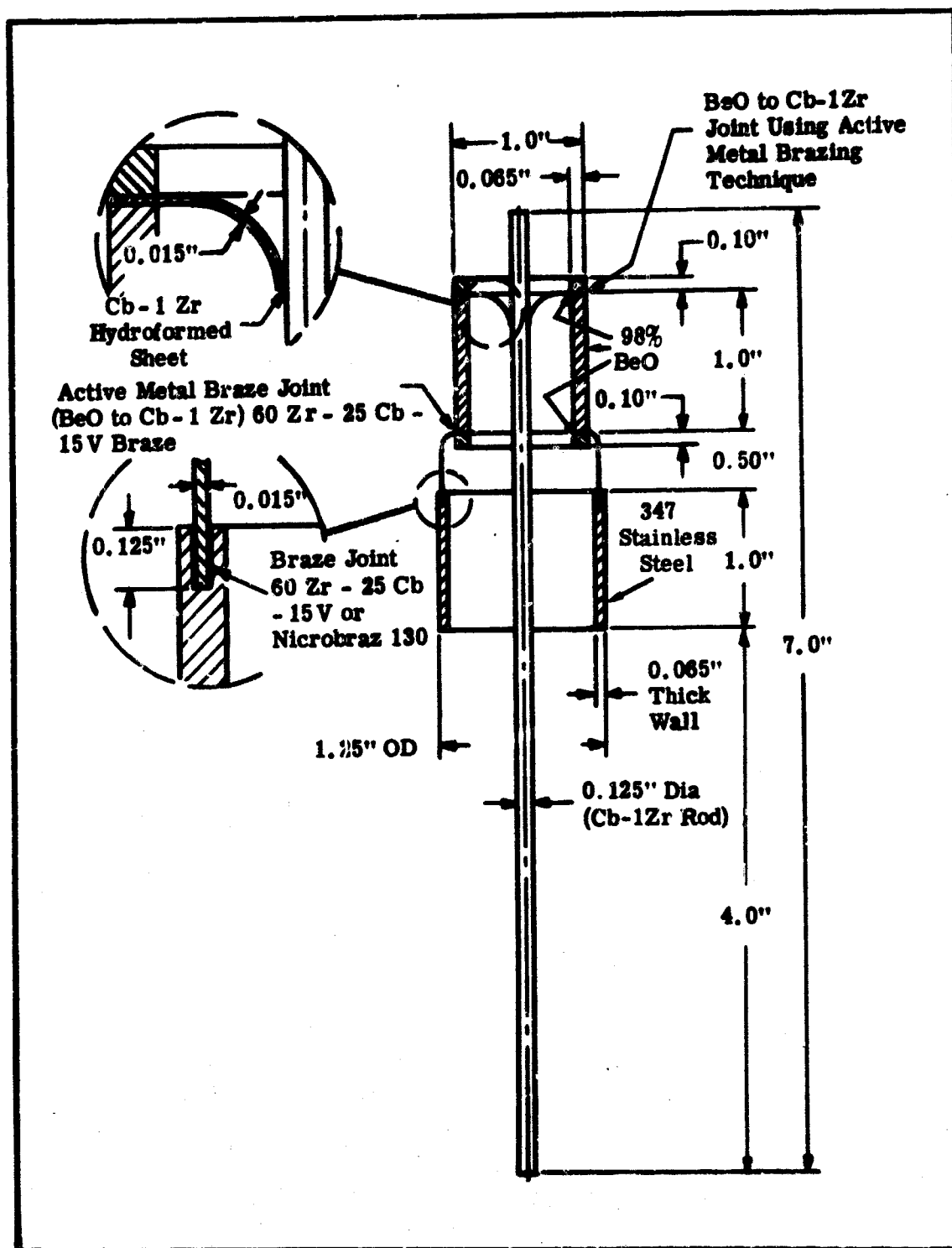


Figure 21. Ceramic-to-Metal Electrical Feedthrough Terminal Seal Configuration

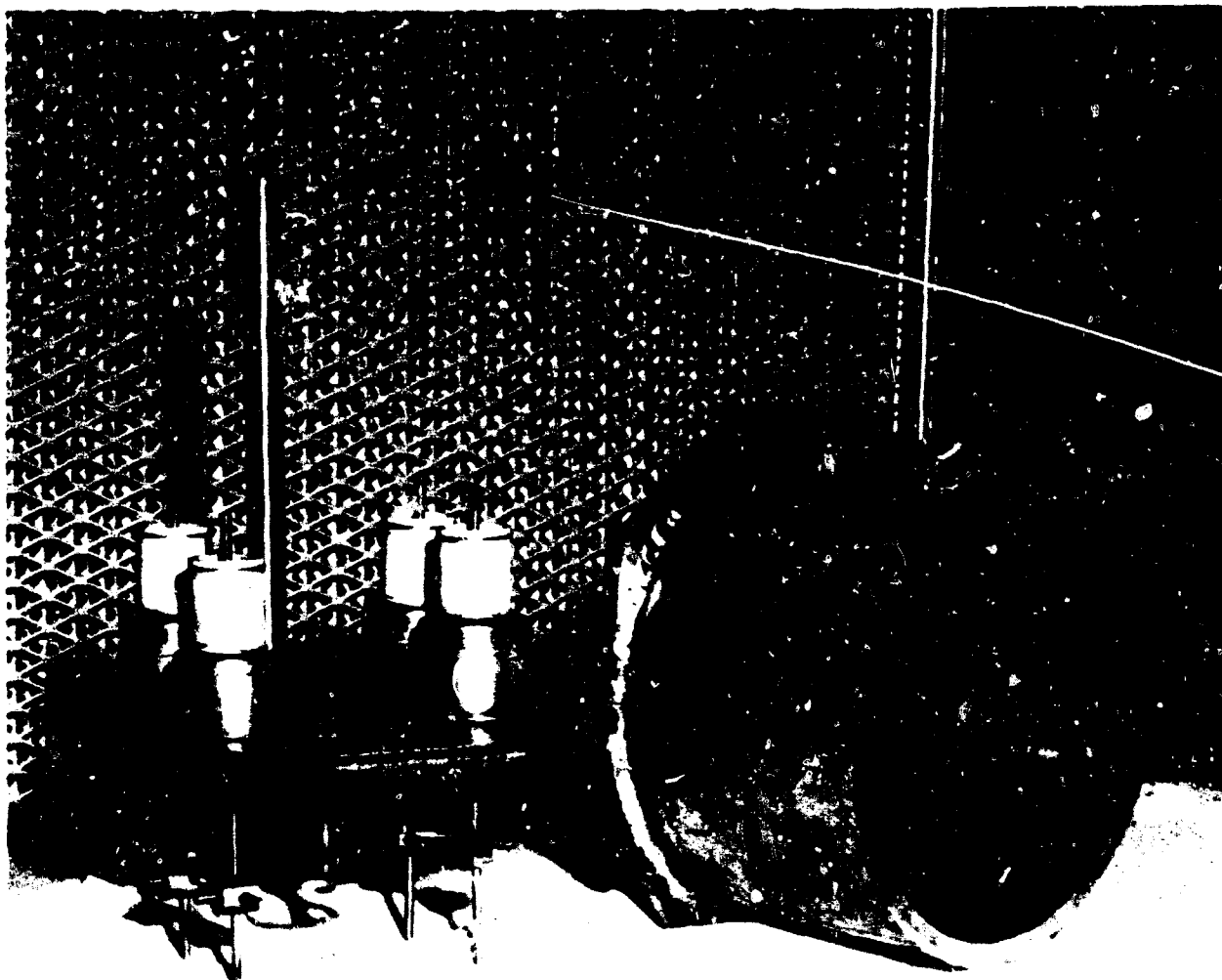


Figure 32. Four-Terminal Conductor Test Container

after assembly, and after the top plate assembly had been heated to 600°C in a vacuum furnace.

(2) Resistance and Resistivity Comparisons of Nickel-Clad Silver Conductors

Total resistance of a conductor circuit can be measured in a two-terminal test container. Resistance of one element in a circuit can be measured in a four-terminal test container. The latter resistance can be expressed as resistivity when the geometry of the circuit element is taken into account. Total circuit resistance measurements were obtained on the two-terminal test container at room temperature and 600°C; both with and without potassium, and under other temperature and heating test conditions. Resistance measurements were made on a nickel-clad silver conductor in the four-terminal container at room and elevated temperatures both with and without potassium vapor. Resistivity values were calculated from the latter resistance measurements. Resistance measurements were made on both containers with a Kelvin bridge using the four point method.

(a) Two-Terminal Test Container

The electrical circuit tested in the two-terminal test container consisted of a measured 12-inch length of nickel-clad silver conductor, two columbium-1% zirconium rods of the feedthrough terminals, and the joints between the rods and the conductor. Two probes were connected to each feedthrough terminal to complete the four terminal Kelvin bridge circuit. Electrical resistance of this circuit was determined in the absence of potassium as the test container was cycled in a vacuum furnace from room temperature to 600°C to room temperature. Figure 33 shows resistance values during this test without potassium.

Preparation for potassium vapor testing consisted of checking the mechanical integrity of the conductor feedthrough joints (they were tight), TIG welding the container parts together, helium leak checking the container (it was leak-tight), loading with approximately two grams of high purity potassium and

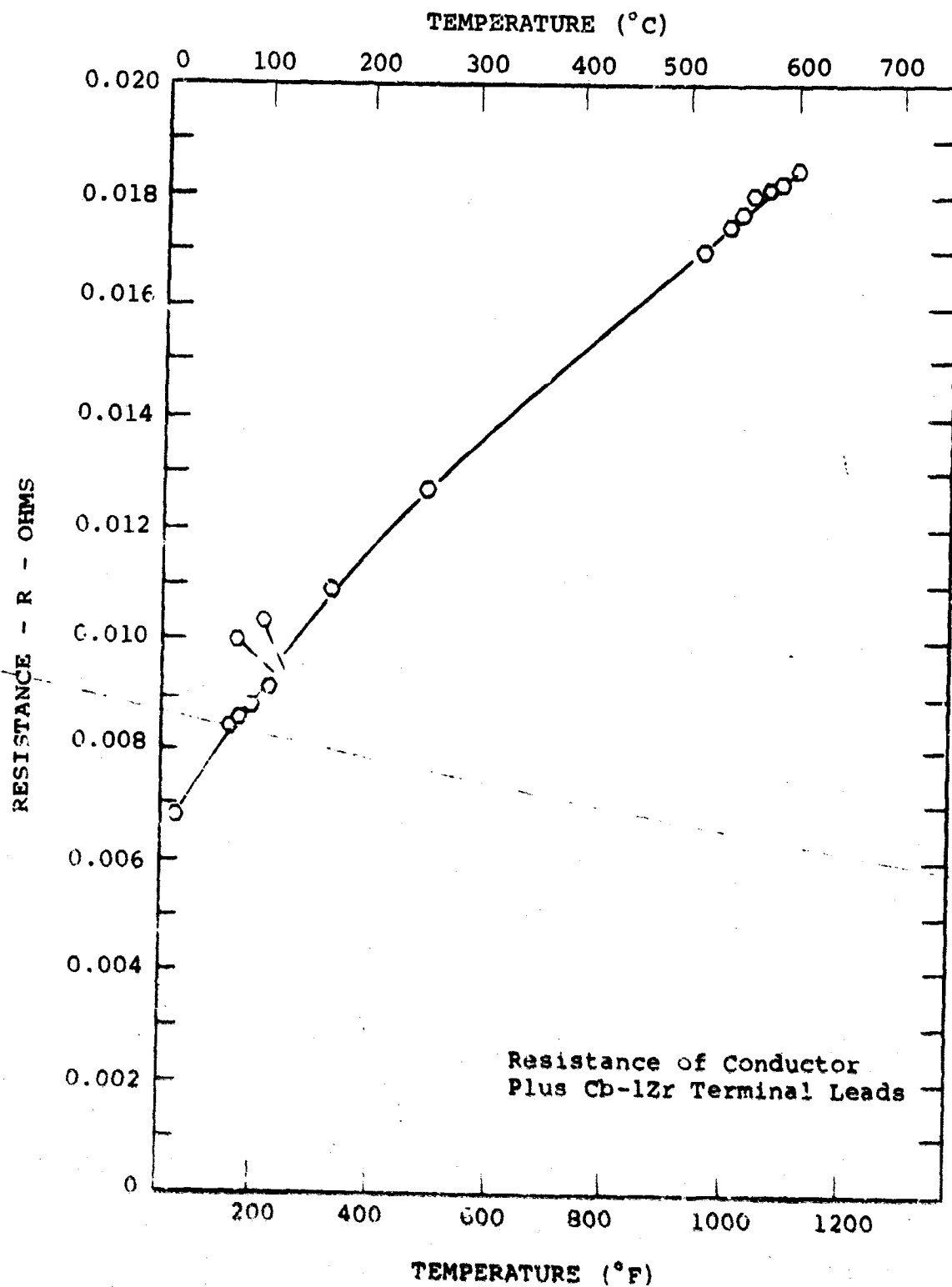


Figure 33. Electrical Resistance-Temperature Relationship, Two-Terminal Test Container Without Potassium

evacuation and sealing. The first potassium test consisted of a temperature cycle from room temperature, to 600°C, to room temperature. Figure 34 shows measured resistance values from this test. The initial resistance (room temperature of 23°C) was higher than that shown in figure 33.

Potassium wetted the ceramic (Lucalox) of the feedthrough terminal at approximately 437°C thus adding another conducting path parallel to that tested earlier. This added path produced a decrease in total circuit resistance (as is shown in figure 34) and persisted through subsequent tests. The same effect was noted previously when a similar electrical terminal seal was tested on Air Force Contract AF33(615)1360⁵.

Electrical breakdown of this Lucalox to columbium-18 zirconium terminal seal occurred at 475°C; however, a steady decrease in the electrical insulation resistance began after a temperature of 250°C was exceeded. The test voltage was 500 V d-c in this previous experiment which would ionize the potassium vapor. The output voltage of the Kelvin bridge which was used to obtain the data in figures 33 and 34 was only 1.5 V d-c so ionization of the potassium vapor probably did not occur.

An attempt was made to eliminate the electrical short circuit (0.3 ohm), by energizing the conductor with a high current from a low voltage source. The two-terminal container was not raised to 600°C with an external source of heat; instead, the temperature was allowed to rise as a result of the electrical input as shown in table XIX. After the 164 minute interval, the test container was allowed to return to room temperature (~25°C). An electrical resistance measurement taken from the terminal seal to the stainless steel container indicated the resistance had increased to 300 ohms. This effect is explained by a partial elimination of the potassium film causing the 0.3 ohm electrical short circuit. The potassium vapor could have boiled off the surface of the Lucalox ceramic portion of the electrical terminal seal giving, in effect, a conductive path from terminal to container with a reduced cross section.

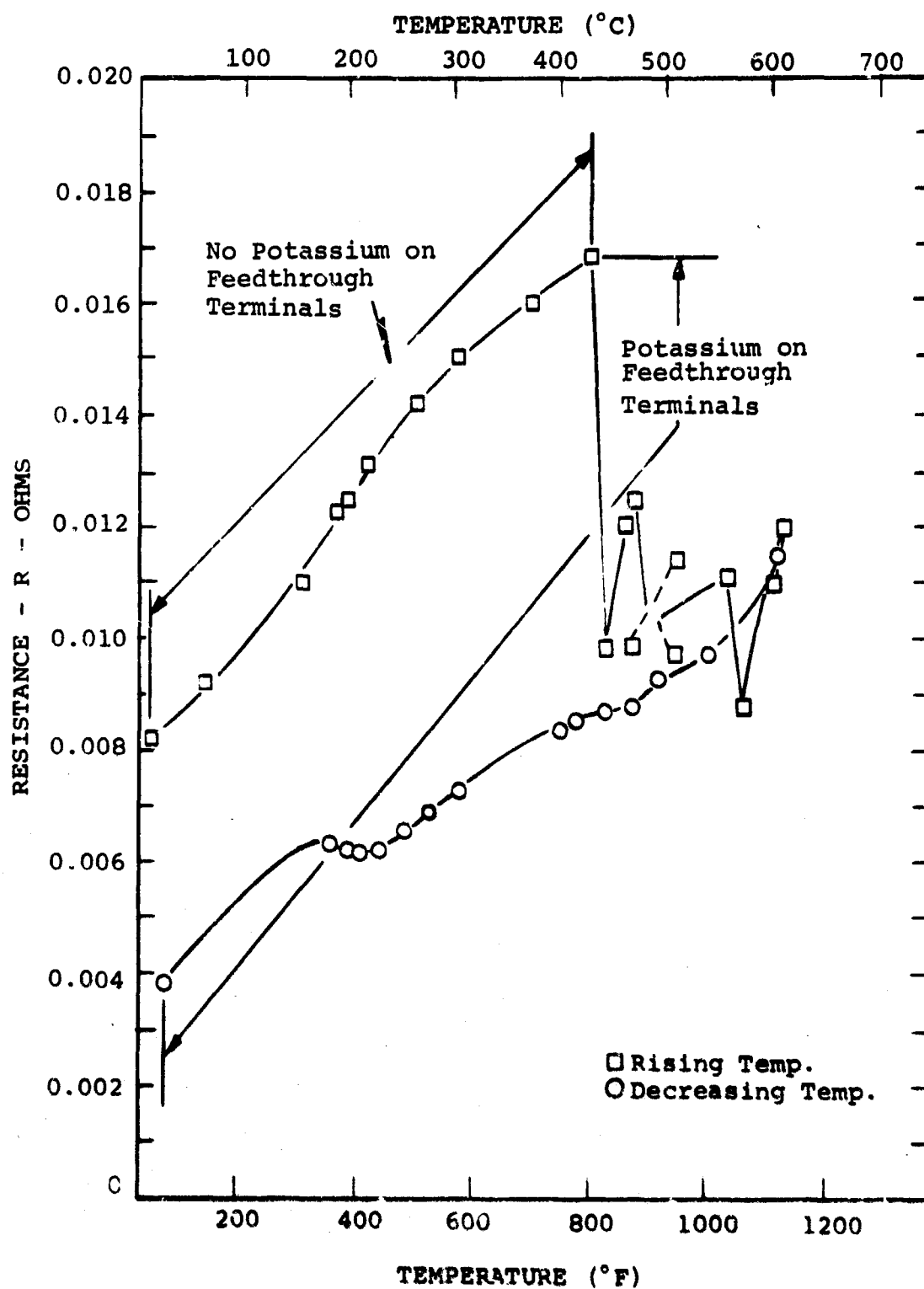


Figure 34. Electrical Resistance-Temperature Relationship, Two-Terminal Test Container With Potassium

TABLE XIX. Temperature Rise of Potassium Vapor Around an Energized Nickel-Clad Silver Conductor

Time (min)	Temperature (°C)	Voltage Input (a-c volts)	Current Input (a-c amps)
0	25	2.13	90
2	34	2.18	81
87	168	2.35	72
101	175	2.34	67
106	183	2.34	67
111	192	2.34	67
164	194	2.34	67

The two-terminal test container was again placed in a vacuum chamber and the chamber evacuated to a pressure of 2×10^{-6} torr. The container terminals were then energized at room temperature from a 2.5-volt, a-c, 90-ampere source. This voltage was impressed across the conductor and the increase in terminal seal temperature was monitored. The potassium metal (which was deposited on the seals in previous tests), clinging to and covering the Lucalox insulation portion of these seals, continued to conduct. When the voltage was applied across the potassium bridging the terminals seals (terminal seal to test container), the seal temperature increased very rapidly. Heating the potassium in this manner was not adequate to completely sever the potassium conduction path. The maximum calculated seal terminal to container resistance, 25,000 ohms, was obtained after a measured 250 volts at a current of 10 milliamperes was impressed for three minutes. At this point, the potassium vapor ionized as was evidenced by a change in oscilloscope traces.

(b) Four-terminal Test Container

Nickel-clad silver was also the conductor material tested in the four-terminal container. The four point Kelvin bridge method was used with a separate terminal for each probe. Current leads were connected near the ends of the test conductor and voltage leads were connected inside the current leads (twelve inches apart). Electrical resistance was determined in the absence of potassium as the test container was cycled from room temperature, to 600°C, to room temperature. Resistivity values were calculated based on conductor cross-section and length, and are shown plotted as a function of temperature in figure 35.

The four-terminal container was processed, as was the two-terminal container, with helium leak checks, TIG welding assembly, potassium loading, evacuation, and sealing. Two thermal cycles (from room temperature, to 600°C, to room temperature) were run on this container. Resistivity measurements from these thermal cycles are shown in figure 35.

In the first potassium heat up, at a temperature of approximately 450°C, the potassium wetted the beryllia insulator portion of the terminal seals (as it had wetted the Lucalox insulator in the terminal seals of the two-terminal seal container) forming a conduction path between seal terminals through the test container. The conduction path at this temperature had a substantially higher resistance than the nickel-clad silver conductor and, therefore, did not appreciably affect the resistance of the test circuit as the temperature was increased to 607°C. After several hours at 607°C, the container was cooled to room temperature, removed from the furnace, and checked for possible potassium leaks. No leaks were found. The resistance from terminals to the test container, measured with a Kelvin bridge, was 0.015 ohm. At this time, feed-through terminal seal heaters and thermocouples were added to the test container.

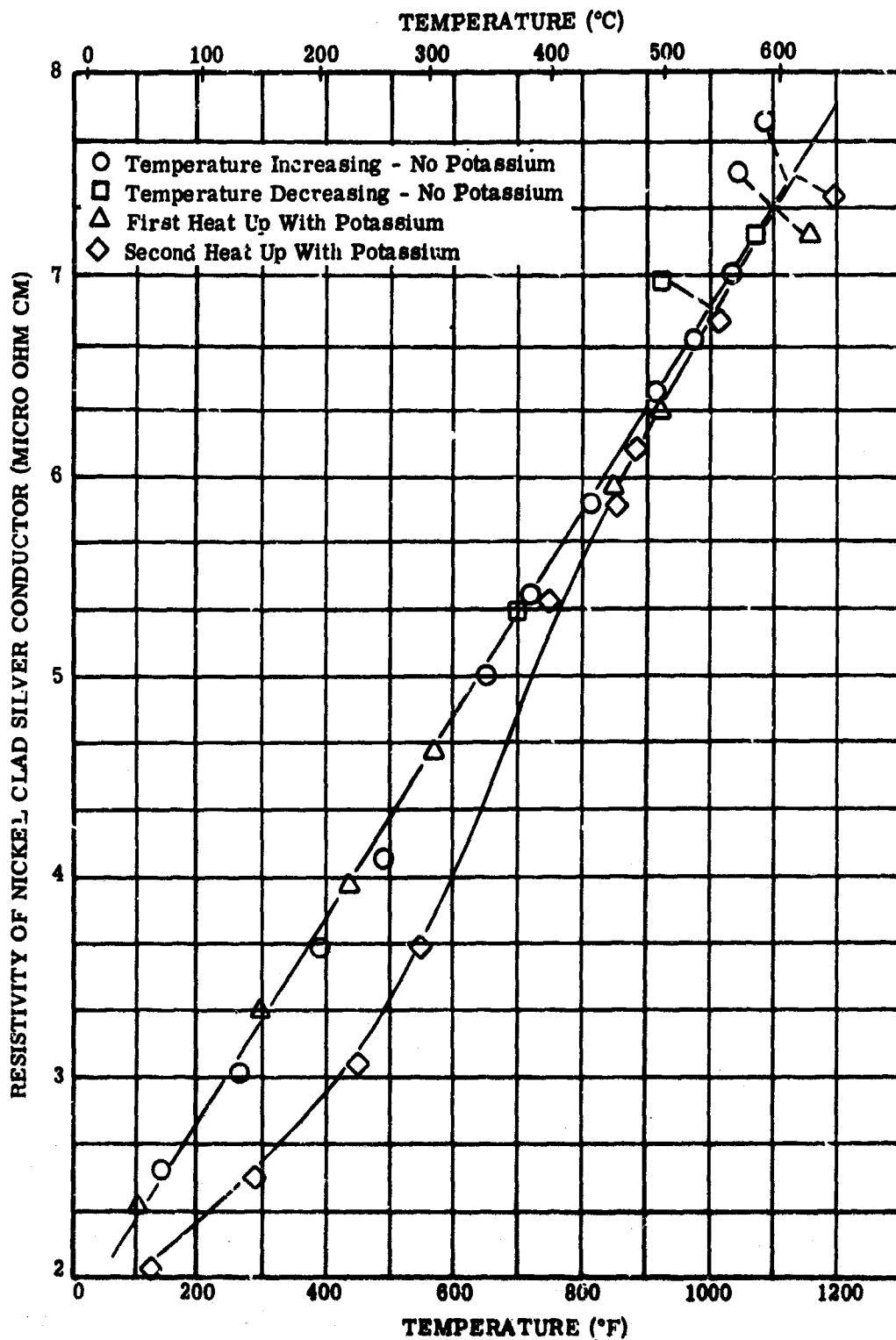


Figure 35. Apparent Resistivity of a Nickel-Clad Silver Conductor as a Function of Temperature with and without Potassium (Four-Terminal Test Container)

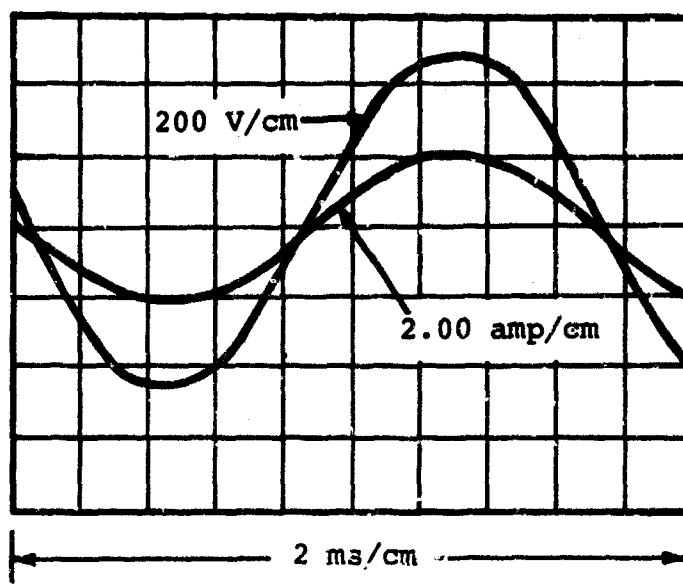
The potassium loaded test container was then heated in the vacuum furnace a second time. The apparent conductor resistance readings varied greatly from the previous readings until a temperature of approximately 450°C was reached. From 450° to 600°C resistance readings were approximately the same as in the no potassium case. Figure 35 shows resistivity during this second heat up with potassium.

The feedthrough terminal heaters were used to raise the temperature of the electrical terminal seals 150°C above the temperature of the potassium vapor as measured in the thermocouple well. The resistance was measured from the terminal to container with the terminal seals at 750°C and the container at 600°C. Terminal seal to container resistance had increased from 22 to 500 ohms with the increase in temperature.

A high impedance power supply was connected across the potassium conductive path from one feedthrough terminal to container.* Potassium on the terminal was vaporized by this power supply and subsequently went to an ionized state. The terminal temperature increased 71°C above the container temperature of 686°C when the vapor ionized. This ionized condition resulted in a voltage reduction across the plasma down to 27.5 volts rms, 60 Hz and a current increase to approximately 2.5 amperes rms. Figure 36 shows the voltage and current before ionization. The terminal temperature dropped as a result of the ionization because the input power was reduced. Figure 37 shows the voltage and current after ionization. (The corresponding temperature drop was from 757° to 637°C.)

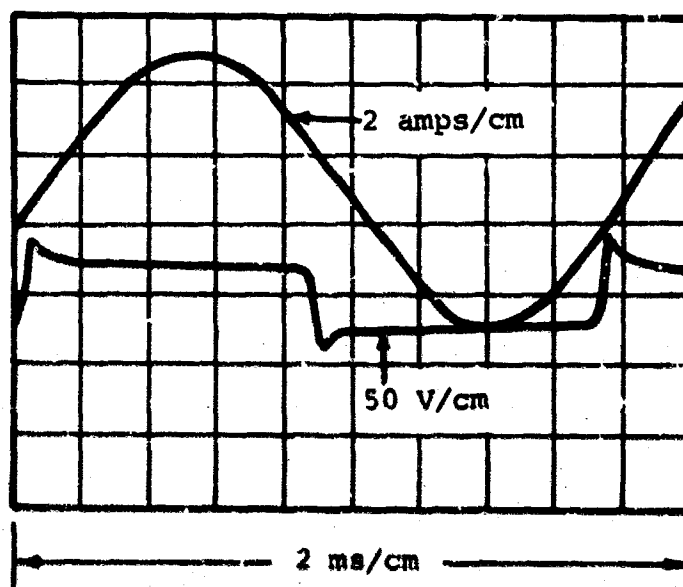
An effect was noted on the potassium plasma when the hot-wall vacuum furnace heating coil was electrically energized and de-energized. The magnetic field generated by the furnace heater winding was sufficient to cause the

* The high impedance supply was a series reactor current-limited supply incorporating a linear a-c reactor similar to that employed in mercury vapor lamp operation. The open circuit-short circuit characteristics of the supply were approximately 440 volts and 5.0 amps rms at 60 Hz.



Power = 440 watts

Figure 36. Traces of Voltage and Current Before Potassium Was Ionized (Four-Terminal Test Container)



Power = 27.5V x 2.5 amps

Figure 37. Traces of Voltage and Current After Potassium Was Ionized (Four-Terminal Test Container)

effective resistance of the plasma to change. The voltage across the plasma, at a constant current, increased from 27.5 volts a-c when the furnace was "on", indicating an increase in effective electrical resistance. This could be explained by the magnetic field causing the plasma to spiral, increasing the mean length of the plasma path, thus increasing its overall effective resistance.

The nickel-clad silver conductor was energized by 80 amps at 0.324 volt for 240 hours. There was no apparent degradation from this test.

The potassium seemed to adhere to the beryllia walls of the terminals by some unknown mechanism. This effect appears to be a function of potassium concentration that is related to volume, vapor pressure, and temperature.

A total of 917 hours was accumulated while the four-terminal test container was at 600°C. Resistance measurements were made on the test conductor at various times during the test. Resistivity values were calculated from these measurements and are shown in figure 38 with the temperature of the conductor at the time of measurement. An attempt was made to correct the resistivity of the conductor to a constant temperature of 1112°F (600°C) by applying a correction factor. This factor is the product of the difference between 1112°F and the test temperature times 5.1×10^{-3} ohm/°F (which is the slope of the upper portion of the curve in figure 35). The squares in figure 38 are the estimated resistivity at 1112°F for the nickel-clad silver conductor. A slight increase in resistivity of the order of 3.5% (based on resistivity at start and end of test) for the 917 hours is shown by the curve.

(3) Evaluation of Test Containers

Test containers, after the completion of testing, were opened, potassium removed, and both container parts and the conductors examined. No evidence of degradation was found after examining the stainless steel parts of the two containers. The Lucalox feedthrough terminals from the two-terminal test

container showed no degradation. One of the four beryllia feedthrough terminals from the four-terminal test container showed the loss of some beryllia from one inside edge. The other beryllia terminals showed no evidence of degradation. The loss of beryllia is probably due to ion erosion encountered when potassium was ionized and sustained current was conducted between the effective terminal seal and the test container.

b. Magnetic Material Test System

A system to continuously monitor magnetic properties of materials in potassium vapor was fabricated, tested, and evaluated. Hiperco 27 alloy Rowland rings, prepared in the same manner as transformer laminations, were tested in this system and were evaluated.

(1) Description of Test Container

(a) General Description

A core box constructed of potassium resistant materials is necessary to contain Rowland rings for magnetic testing in the presence of potassium vapor. One element in the core box must be an electrical insulator to break the electrical circuit that would be created around the Rowland rings if the core box was constructed entirely of electrical conductor materials. Several core box designs using columbium-18 zirconium metal and high alumina ceramic insulating materials were evaluated. A terminal seal type core box was selected for use because of the availability of developed technology* and materials.

(b) Assembly of the Terminal Seal Type Core Box

Major components (except the 1/2-inch diameter fill and evacuation tube and a spacer) for the terminal seal type core box are shown in figure 39. Columbium-18 zirconium sheet 0.030-inch-thick was used for the ends and the outer

* See Section III.A.4.a. for a discussion of terminal seal technology.

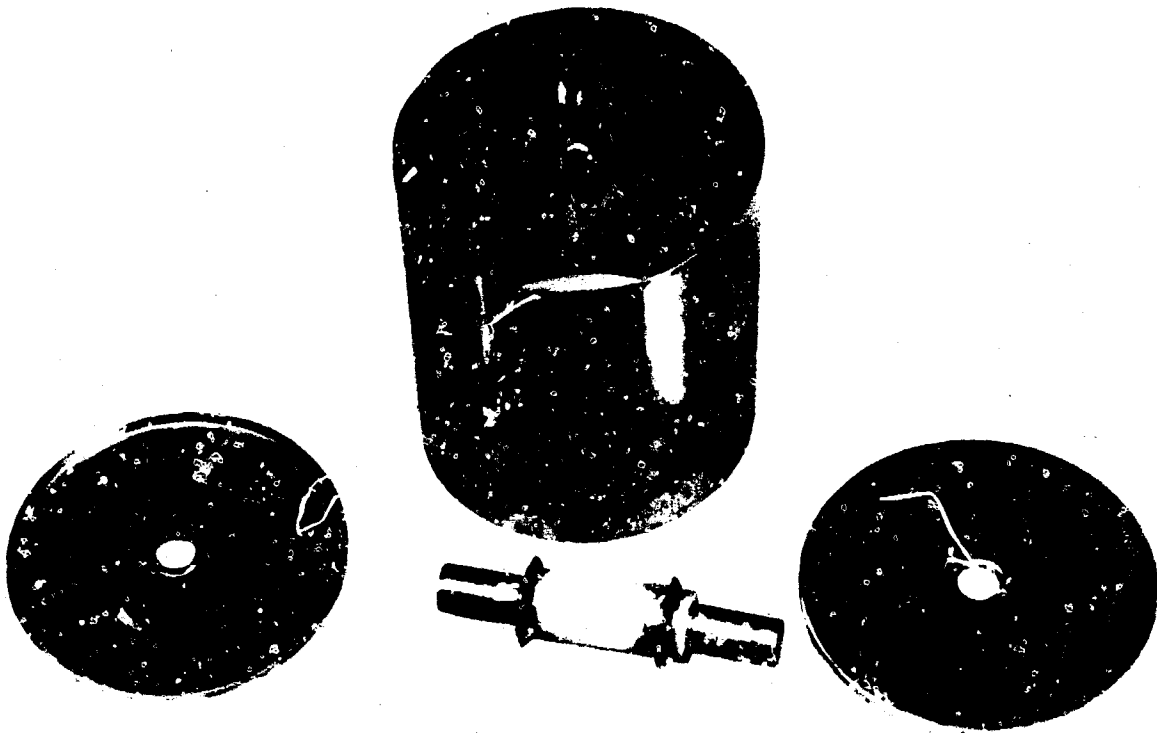


Figure 39. Terminal Seal Type Core Box Parts

cylinder. Prior to assembly, these formed sheet pieces were vacuum annealed at 2300°F for one hour at a pressure of 1.5×10^{-6} torr.

Assembly began by TIG welding one end of the terminal seal tube into an end plate, welding this end plate to the outer cylinder, and welding a 0.500-inch outside diameter fill and evacuation tube into the other end plate. All welding was done in an inert atmosphere glove box with a gas purification system maintaining the oxygen level less than 1 ppm and the moisture level less than 4 ppm. Selected Rowland rings were stacked in the core box so that interlaminar insulation separated each ring. The stack was centered by hot-pressed alumina-yttria spacers placed between the stack and the inside of the core box outer cylinder with the spacers rising above the stack.

The Rowland ring stack was held axially in place (electrically insulated from the columbium-1% zirconium can) and compressed to approximately 1.1-inch high by a cylindrical spacer of 0.030-inch-thick columbium-1% zirconium. Final closure, end plate to outer cylinder and terminal seal tube, was made by TIG welding in an inert atmosphere glove box as before. Parts were fixtured for welding using "C" clamps to hold the end plate in place thus compressing the Rowland ring stack. Figure 40 shows the Rowland core box as assembled at that time.

The quantity of Hiperco 27 alloy magnetic material determined the requirements for electrical excitation and sensing during magnetic testing. Limited area in the center of the core box directed the use of a relatively small number of turns, thereby increasing the ratio of conductor area to insulation area. The following characteristics describe the Rowland ring core:

Rowland Ring Physical Data:

No. of Rowland Rings - 110
Inside Diameter - 3.256 inches
Outside Diameter - 3.890 inches
Magnetic Material Theoretical Stack
Height - 0.88 inch

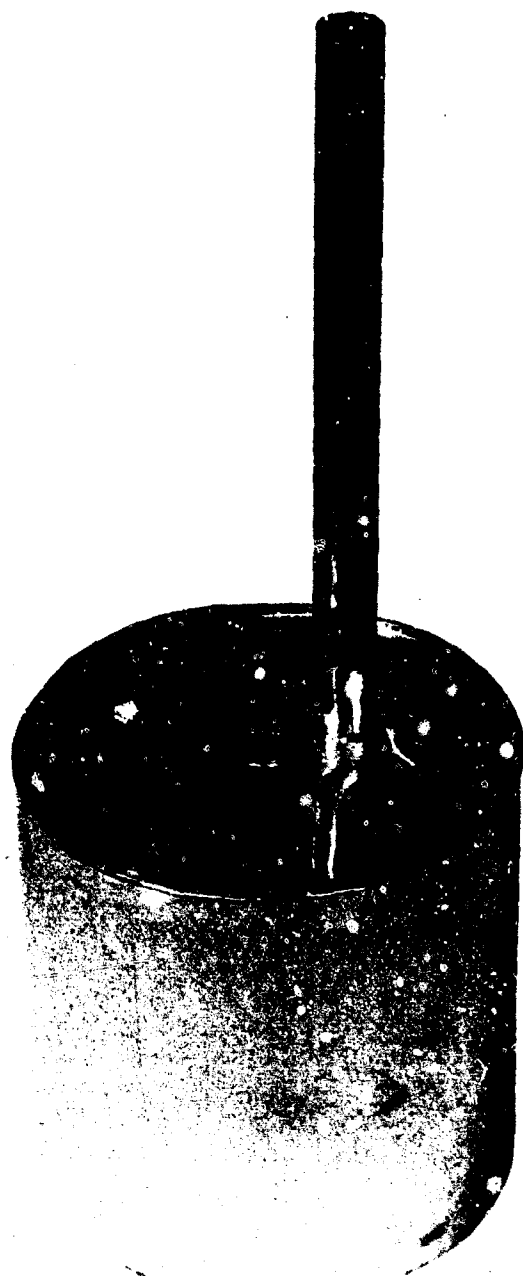


Figure 40. Weldsa Rowland Ring Core Box

Weight - 0.912 lbs.
Density - 2.85 lbs./cu. in.

Rowland Ring Mean Magnetic Path:

Length - 11.2 inches
Core Cross Section - 1.79 in.²

The copper conductors for the primary and secondary windings were insulated with Re-frasil, a woven sleeving of vitreous fibers (99% SiO₂). Five turns of No. 10 wire were used for the primary winding and two turns of No. 21 wire were used for the secondary winding. The individual conductor turns were joined to form windings by TIG welding in the argon atmosphere glove box. A mock-up of core box windings was made using the same conductor materials, fabrication techniques, and geometrical relations as the Rowland ring core box. This mock-up winding, enclosing no magnetic material, was used to establish the air inductance of the windings. Figure 41 shows the Rowland ring core box as wound.

(2) Changes in Magnetic Properties of Hiperco 27 Alloy

The completed core box was installed in a vacuum furnace for magnetic testing. Braided copper conductors were run from feedthrough terminals on the furnace faceplate to the core box primary and secondary winding terminations. These conductors were electrically insulated with Vycor tubing and, when parallel, were shielded by tubular stranded copper shielding which was grounded to the furnace. Shielded thermocouples were placed in the core box center hole with the windings and on the outside of the core box in the region of the Rowland rings.

Magnetic tests were run in the hot-wall vacuum furnace at 130° and 600°C. The 130°C tests were at a furnace pressure of approximately 4×10^{-6} torr. A vacuum of approximately 2×10^{-6} torr was used during the 600°C test to maintain oxygen at a level that would not attack the columbium-1% zirconium of the core box. The 130°C test temperature was the equilibrium temperature for the core box heated by electrical losses.

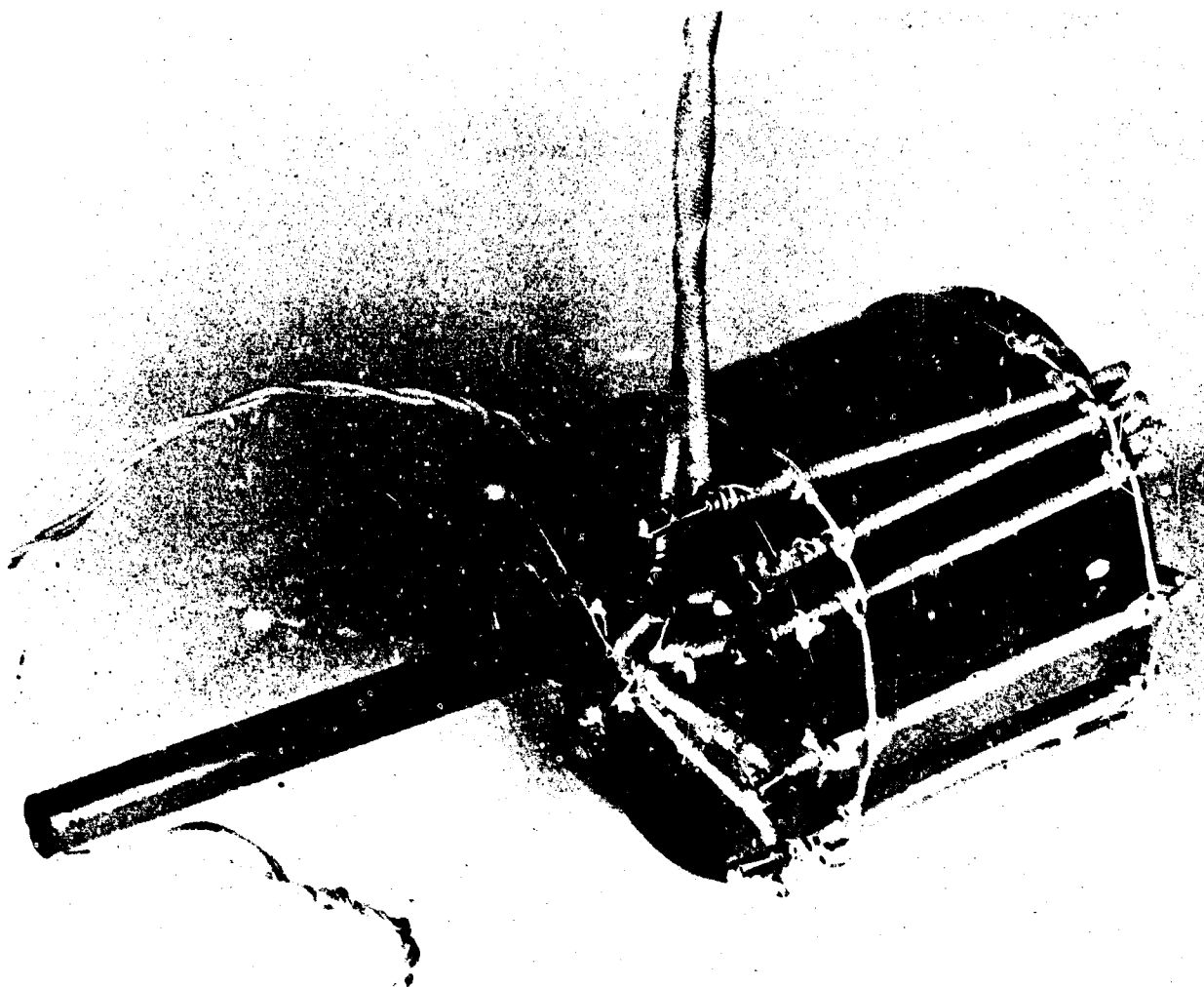


Figure 41. Wound Rowland Ring Core Box

Alternating current core loss was obtained at 400 Hz with sinusoidal flux variation. Measurements were made with the furnace power off to eliminate the magnetic field of the furnace. Figure 42 shows a-c core loss results from these tests and those from a previous test with Mylar interlaminar insulation. The Hiperco 27 alloy Rowland rings with plasma-arc sprayed alumina interlaminar insulation are shown to have a lower core loss than those with Mylar interlaminar insulation. The higher temperature curve shows lower core losses especially at the higher flux densities.

After magnetic tests without potassium the core box was loaded with approximately two grams of potassium. Magnetic tests were conducted at various temperatures.

The effect on the magnetic properties of Hiperco 27 alloy, of saturated potassium vapor is also shown by the core loss curve of figure 42. At the top temperature shown (238°C), the potassium vapor completely short circuited the ceramic Lucalox insulator. This short circuiting turn caused the output of the two turn secondary or sensing winding to fall essentially to zero. No further magnetic a-c core loss data could be obtained.

D-C magnetization tests indicated the core would still function. The conclusion from this magnetic test data is that a laminated core of magnetic material, if insulated with a 99.9% alumina plasma arc sprayed coating 0.5-mils thick, will function with increased losses in the presence of 600°C potassium vapor.

(3) Evaluation of Erosion

Figure 43 shows the severe erosion of the outer edge of a Hiperco 27 alloy Rowland ring specimen after being subjected to the combined effects of a magnetic flux from 500 to 11,500 gauss and 24 to 600°C potassium vapor.

The eddy currents in the Hiperco 27 alloy are apparently diverted from their normal direction and follow the moving potassium conductor on the

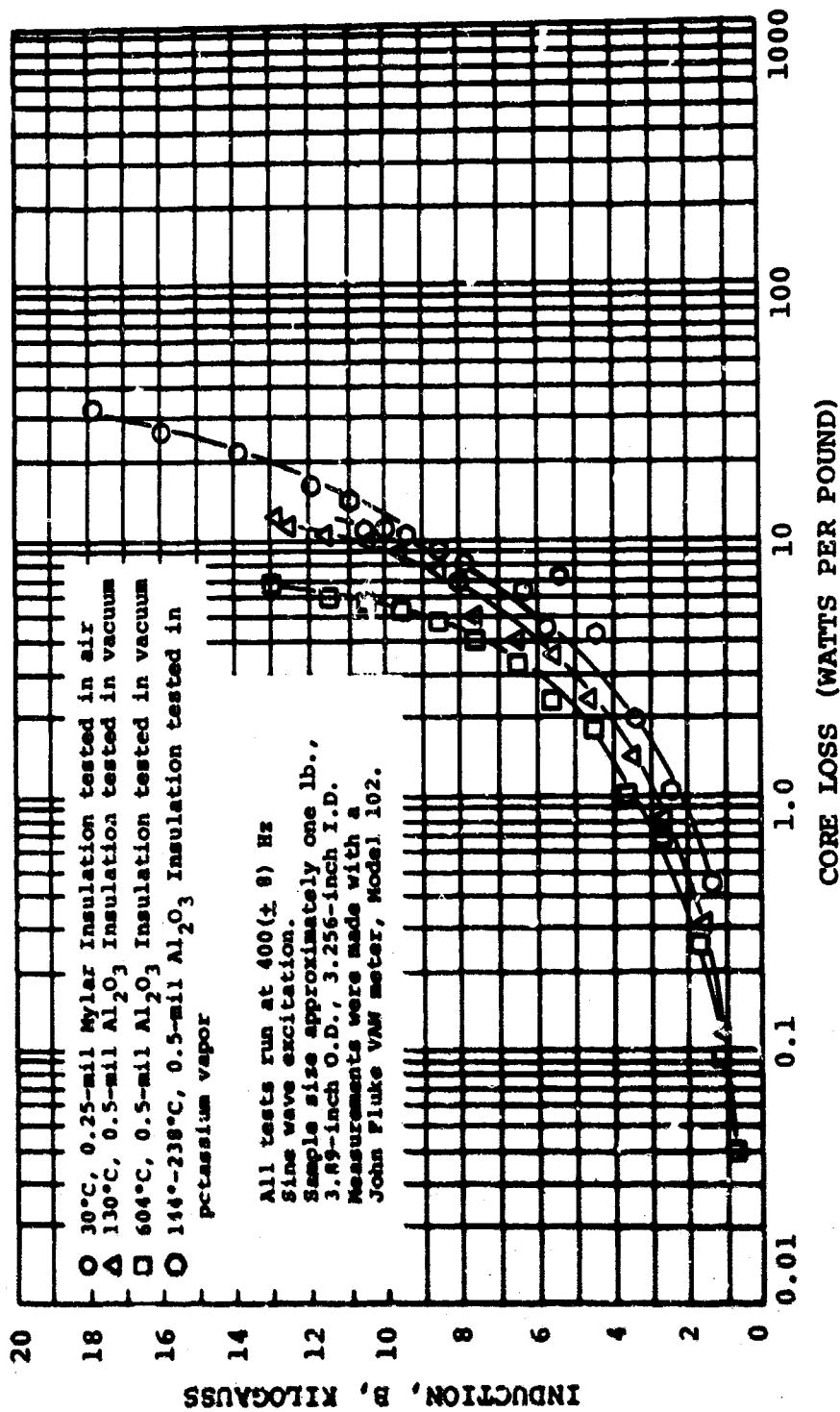
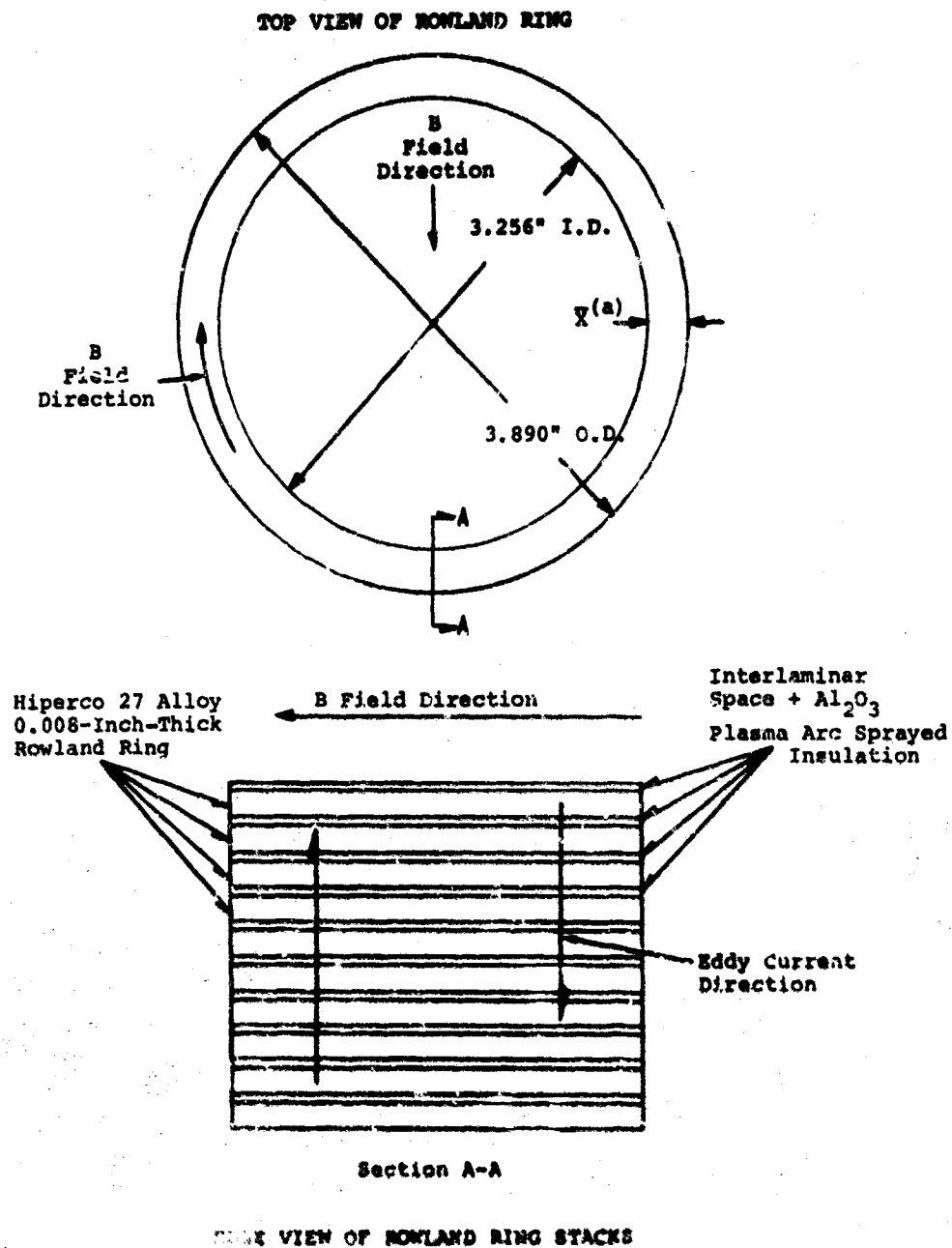


Figure 42. Core Loss, P_c , Annealed 0.008-Inch-Thick Hiperco 27 Alloy Rowland Rings





(a) Constant direction of force exerted on Potassium by

Figure 44. Force and Direction of Potassium in a Rowland Ring Magnetic Field

The previous equation considers the electrostatic field \bar{E} . For practical purposes in the core box, \bar{E} is very small; it may, therefore, be neglected to give:

$$\bar{F} = q(\bar{V} \times \bar{B})$$

The increase in the force driving the potassium to the outer edges of the Rowland ring specimens, therefore, can be considered as increasing directly with an increase in the "B" field strength. The "B" field is changing direction 400 times per second which imparts a vibratory stop-start motion to the potassium film formed between the Rowland ring laminations by capillary attraction causing erosion of the metal.

B. PROCESSES AND TECHNIQUES INVESTIGATED FOR FABRICATION OF POTASSIUM VAPOR RESISTANT ELECTRICAL COMPONENTS

Ceramic and ceramic-metal combinations, processed or applied in different ways, were tested and evaluated for use in potassium vapor resistant electrical devices. Ceramic materials included high alumina compositions, alumina yttria eutectic composition, rare earth oxides, and some commercially available ceramics. Metals used as substrates and in ceramic-metal composites included rhodium, iridium, Hiperco 27 alloy, and nickel-clad silver. Binder materials, used between metals and ceramics, included zirconium and titanium. Ceramic-to-metal applications were made by radio frequency (rf) sputtering, plasma-arc spraying, or slip-coating and heating.

1. General Description of Equipment and Processes

Several equipments and processes were common to making experimental ceramic modulus-of-rupture bars and ceramic/metal fabrications. General descriptions of these equipments and processes follow.

a. Hot Pressing

Ceramic and ceramic/metal composite discs were made from prepared powders by cold pressing followed by hot pressing. Ceramic powders were prepared for pressing by sizing (using only those which would pass through a 325 mesh screen) and drying for two hours at 125°C in an air oven. A measured amount of powder was then cold pressed. The cold pressing die was steel and was 0.005-inch smaller in diameter than the hot-press die.

Powders were separated from steel punch faces by Glassene (a paper coated with a bond release agent). All cold pressings were made with a force of 20 tons per square inch and were followed by drying at 125°C for 2 hours in an air furnace.

Hot pressing was performed using the equipment shown in figure 45. This equipment included a 20-kW rf generator operating at a nominal frequency of 450 kHz. Power from this generator induction heated the cold pressed disc through the use of graphite susceptors which were also the hot pressing die and punches. The die was a "floating" die (i.e. floated by being supported by springs) with a through hole and punches entering from both top and bottom. Figure 46 is a schematic of the hot-press "floating" die assembly. The "floated" die approach gave a more uniform density distribution than a fixed die, as was established earlier⁵. Die punches were 2.000 inches in diameter and fit in the die with 0.003-inch diametrical clearance. Spacers of HP Grafoil 0.010-inch thick were used between punch faces and the ceramic disc to prevent the ceramic from bonding to or chemically reacting with the graphite punches. Pressure supplied by a hydraulic jack, was applied to the graphite punches through back-up ceramic punches and the press frame. Hot pressing was done under a cover of argon which was channeled through the die assembly as is shown in figure 46.

The initial steps used in hot pressing were common to all materials. After the die assembly was loaded with a cold pressed disc it was purged with argon for 30 minutes. A load of 325 psi was then applied and rf heating begun. This load was maintained as the die assembly was heated to 2300°C (as determined with a "Pyro" brand optical pyrometer). The pressure was then increased to 5000 psi and heating continued until a temperature in the range from 2500° to 2700°F was reached. At constant temperature and pressure the change in thickness of the compact was measured over a period of time from about 15 to 30 minutes. When the rate of change in thickness began to approach zero the temperature was raised an additional 25° to 50°F and held for another 10 to 15 minutes. If the thickness decreased substantially after this temperature increase a longer holding time was employed or the temperature was raised an additional increment until no further decrease in thickness could be observed. All materials processed in this manner had high densities and fine grained microstructures.

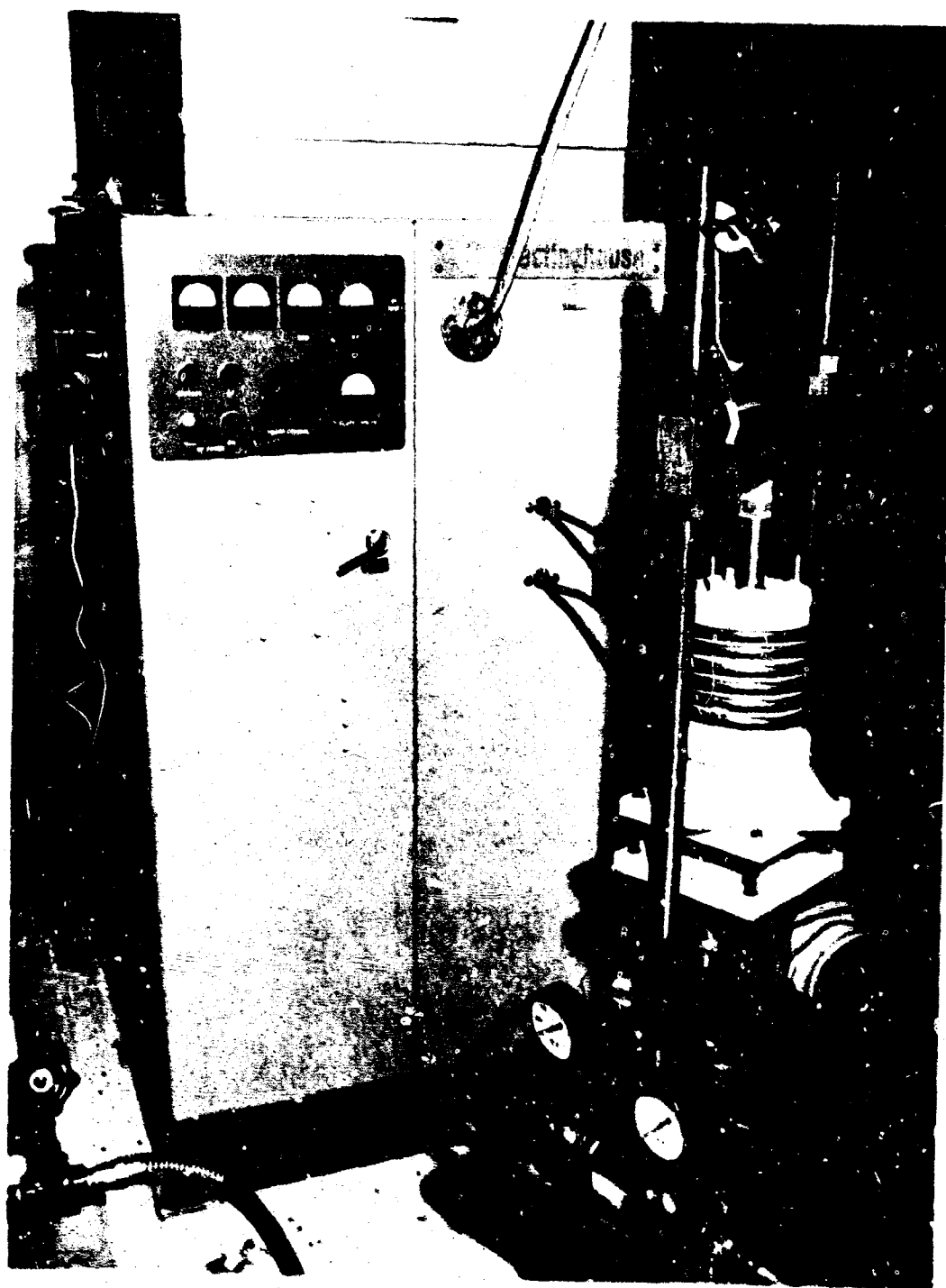


Figure 45. "Floating" Die Hot-Press Assembly and RF Generator

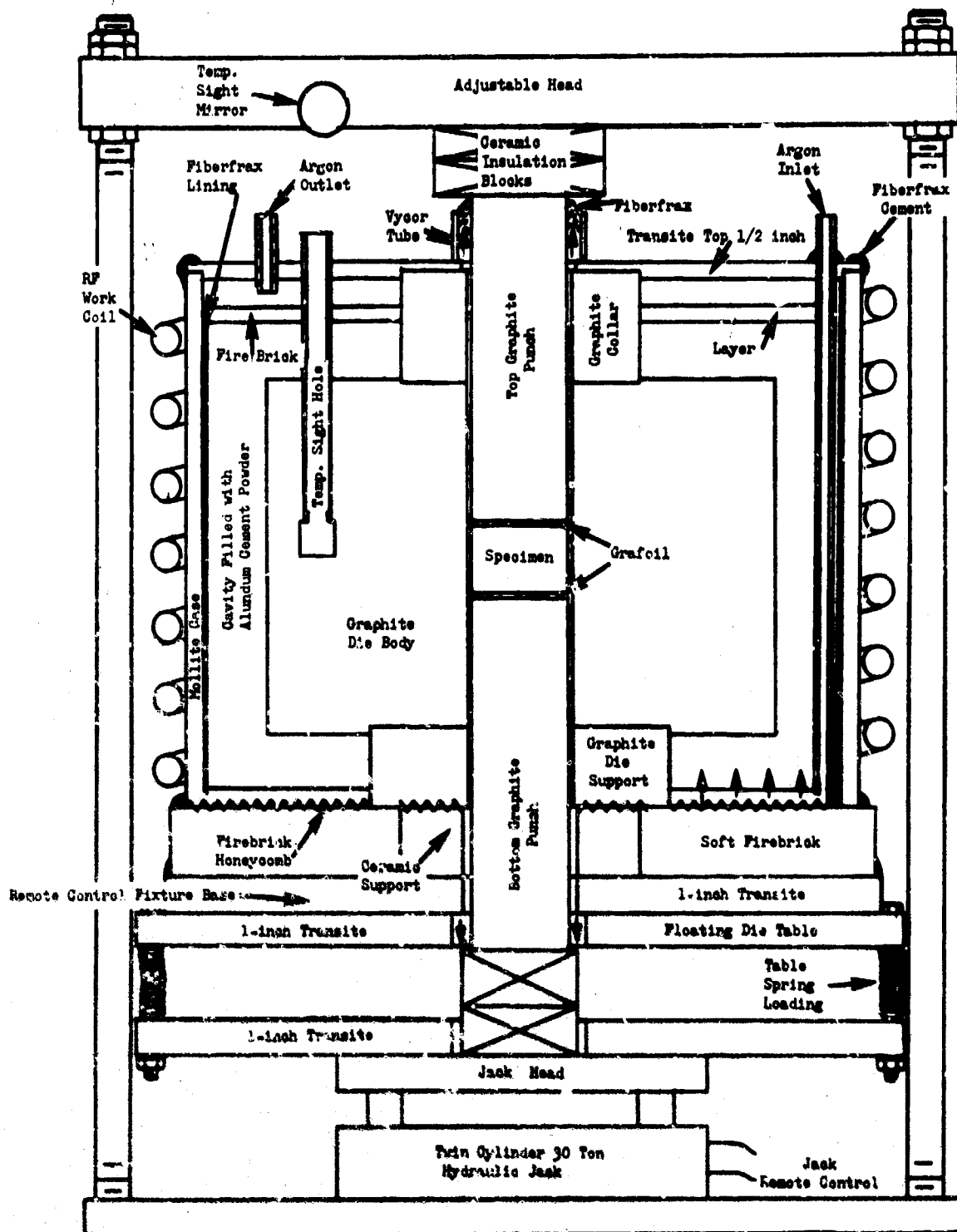


Figure 46. Hot-Press Floating Die Assembly Schematic

b. Radio Frequency Sputtering

Thin films of metals and oxides were deposited on various substrates using the Consolidated Vacuum Corporation sputtering system shown in figure 47. The rf power supply (AST-200) was used to sputter both metals and insulations. The bell jar was pumped by an oil diffusion pump through a liquid nitrogen trap. Sputtered specimens were prepared by rf techniques using a "diode" target configuration. A typical rf target arrangement is shown in figure 48. The target disc was either a metal or a ceramic insulator which was electrically connected to the output of the rf power supply. Radio frequency power at 13.56 MHz was applied to an electrode on the back side of an insulating disc or directly to a metal disc depending on the material to be sputtered. A negative d-c bias voltage was developed on the target. Positive argon ions which were created in the plasma surrounding the target then bombard the surface of the target and sputtering was initiated.

A typical sputtering cycle began with pumping the chamber to the 10^{-6} torr range and then backfilling with argon to a pressure in the range from 5 to 7 microns. An argon plasma was then created in the target region by application of rf energy. A magnet coil that encircled the bell jar was used to generate a magnetic field. When the intensity of this magnetic field was increased by increasing the d-c current in the coil, the plasma density and hence sputtering rate was increased.

c. Plasma-Arc Spraying

Plasma-arc spraying on previous programs^{5,6} was done with a hand held plasma-arc torch and with the substrate in air. The resulting coatings had acceptable uniformity, but the substrates were oxidized in the region where ceramic particles were deposited. Oxides of Hiperco 27 alloy are not resistant to potassium vapor, thus, the plasma-arc sprayed ceramic inter-laminar insulation was not stable in the 600°C potassium environment.

Plasma-arc spraying on this program was done using a controlled atmosphere glove-box system which featured automatic traversing for the spray pattern. A view of one portion of this system is shown in figure 49. The chamber was three feet in diameter and five feet long.

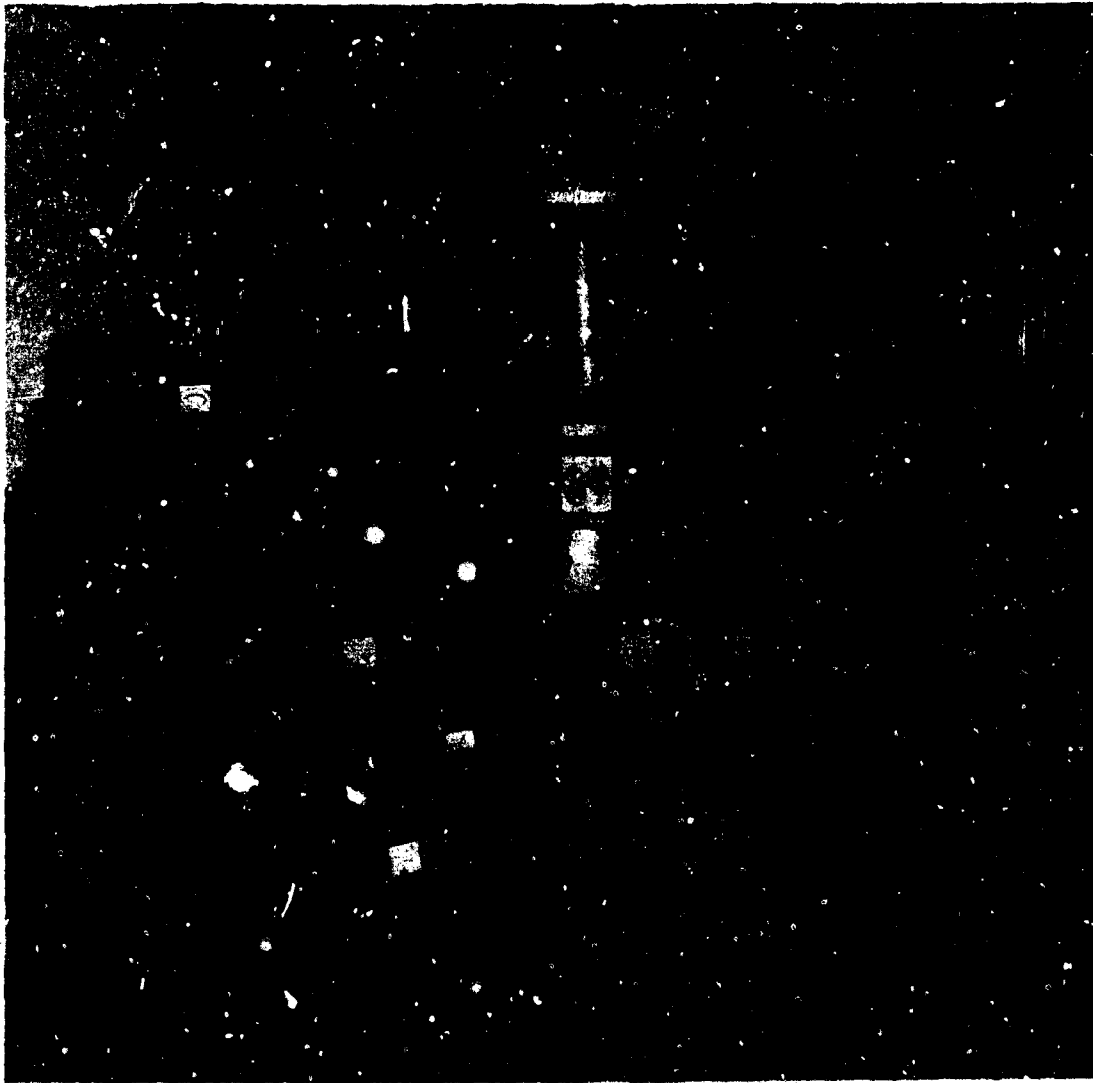


Figure 47. RF Sputtering System

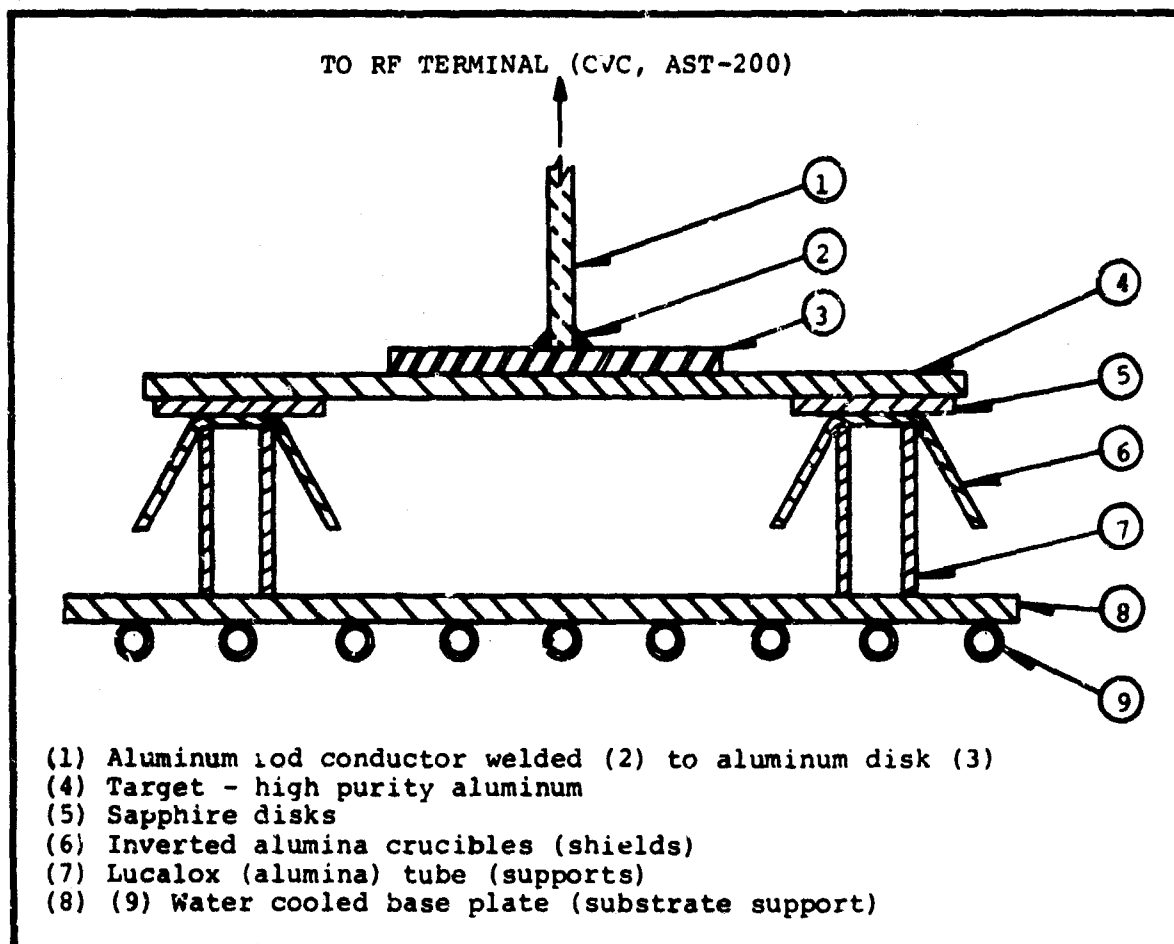


Figure 48. Sectional View of RF Sputtering (diode) Assembly

An evacuation subsystem, using both mechanical and oil diffusion pumps, evacuated the chamber to a pressure in the 10^{-4} torr range after each exposure of the chamber interior to air. This pumping subsystem was also used to evacuate the antechamber through which substrates were transferred. An argon supply and conditioning subsystem furnished argon for both the plasma-arc spray torch (from high pressure cylinders) and the chamber atmosphere (from high pressure cylinders or lower pressure liquid tanks). Argon from the chamber

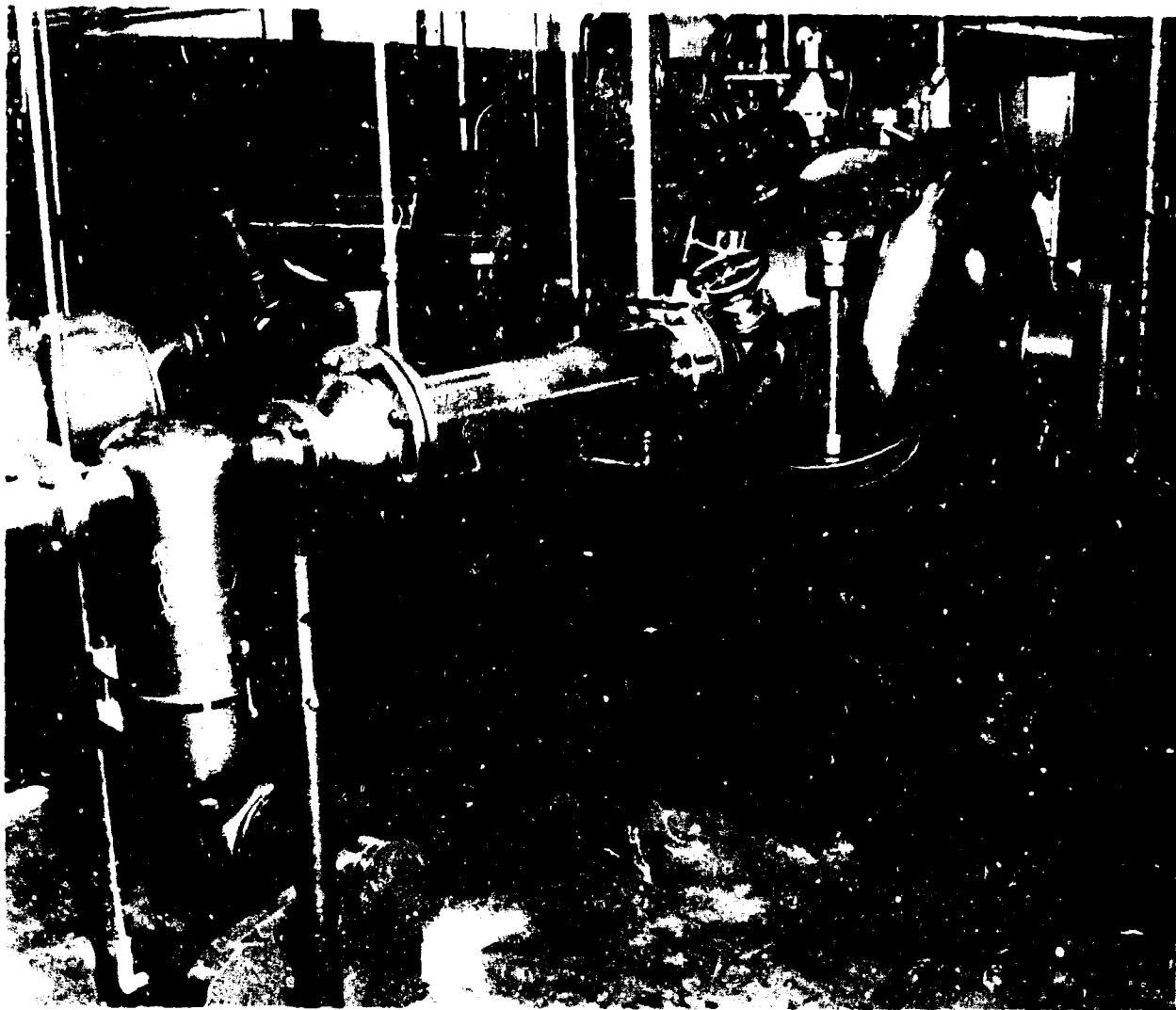


Figure 49. Plasma-Arc Spray Controlled Atmosphere System

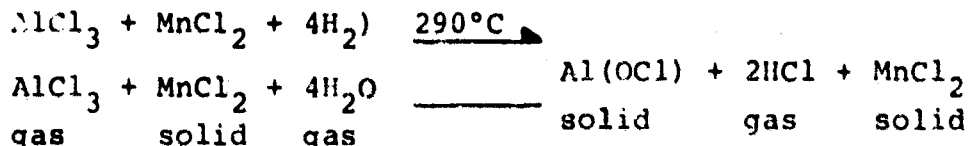
was conditioned by a heat exchanger which cooled the gas and was circulated through a series of two filters which removed dust. Oxygen and moisture monitors were used to monitor the quality of the effluent gas from the chamber.

Three degrees of freedom were provided the torch-work piece combination. The torch was positioned vertically in the chamber by mechanical means and was translated horizontally in programmed steps in the axial direction using hydraulic power. A work-piece table was oscillated horizontally at an adjustable rate across the chamber, also using hydraulic power. The work-piece table was made of 1/4-inch-thick copper plate with copper tubing soldered to its bottom side. Cooling water was circulated through the tubing, thus cooling the table. Flexible water lines connected the movable table to the chamber's water system. Magnets were also fixed to the bottom side of the work table and were used to hold in place the material to be sprayed. The plasma-arc spray subsystem consisted of a Plasmadyne Model PS-62 power supply, Thermal Dynamics Model PI-100-1 powder feeder, and a Thermal Dynamics Model H-50 plasma torch equipped with a model 57x30 spray holder, a 57x34 insert and a 57x2 electrode.

d. Compounding of Aluminum Oxychloride

One way of depositing high purity alumina on high temperature materials is to thermally decompose aluminum oxychloride in an air or hydrogen atmosphere. High-purity aluminum oxychloride was not available on the open market so one batch was prepared at Westinghouse.

Aluminum Oxychloride was prepared using a modification of a method published by Lyu and Zvyagintsev⁷. The pressure and temperature during the reaction were recorded as a function of time and are shown in table XX. Figure 50 shows the temperature instrumented stainless steel reaction vessel in which aluminum oxychloride was made. The simplified reaction equation is as follows:



The starting materials, aluminum chloride 99+% and manganese chloride tetra-hydrate, were weighed into separate high purity alumina (99.5%) tall form crucibles.

TABLE XX. Pressures and Temperatures During Aluminum Oxychloride Preparation

Time (minutes)	Container Outside (temp. ° C)	Reaction Area (temp. ° C)	Press (psig)
0	435	100	35
3	435	150	52
5	430	170	52
8	412	175	69
10	399	180	72
		Power Off	
15	379	185	74
20	362	190	74
25	352	190	73
30	335	185	72
35	319	160	71
		Power On	
40	336	180	71
		Power Off	
45	393	180	72
50	385	185	73
55	378	185	73
60	366	190	73
65	351	190	73
70	337	185	71
75	324	180	70
		Power On	
85	398	190	70
90	444	200	72
98	434	210	74
105	500	250	76
112	574	270	82
115	612	275	85
120	612	280	89
125	595	285	90
130	575	290	90
135	547	290	89
140	514	280	86
145	498	280	84
150	478	280	81
170	498	280	78
205	486	280	78
225	528	280	77
245	486	280	75
252	458	270	72
262	512	280	74
290	498	270	67
305	549	280	71
320	476	280	71
335	495	270	69
350	481	280	69
		Power Off	

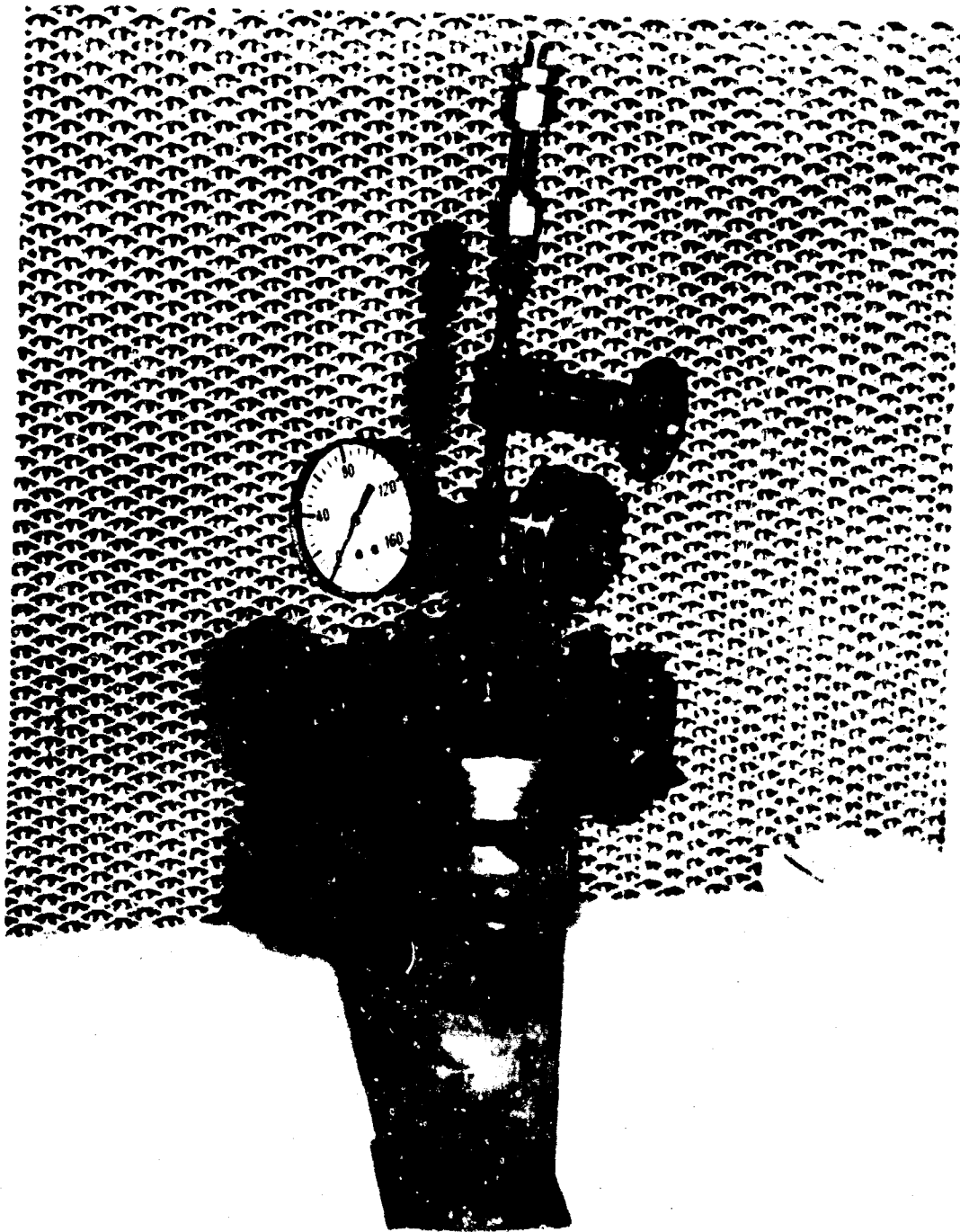


Figure 50. Aluminum Oxychloride Reaction Vessel

These crucibles are shown in figure 51 as they appeared after aluminum oxychloride preparation. The aluminum chloride was introduced into the larger alumina container in a 10.7 gram quantity. Four grams of manganese chloride tetra-hydrate were weighed into the medium size alumina container which was then set inside the larger container on a high purity alumina spacer which is shown as the smallest crucible in figure 51. This arrangement effectively prevented mixing and contamination of the desired product with manganese chloride. The manganese chloride tetra-hydrate was used in the reaction to supply water at a slow rate. This prevented an instantaneous reaction which could possibly lead to hydrolysis of the aluminum oxychloride giving aluminum hydroxide as the end product.

The percent yield of aluminum oxychloride was 75% of theoretical. This yield, that is 40% greater than that reported in the referenced paper, was possibly due to a change in the reaction conditions caused by carrying out the reaction in high purity alumina crucibles rather than impure ceramic or glass crucibles.

e. Processing Fused Alumina - Yttria Eutectic

Alumina-yttria eutectic was investigated as a slip cast and fired insulation for rhodium and iridium wire, and as a castable ceramic insulation. Aluminum oxide- yttrium oxide eutectic consists of 82 mole percent Al_2O_3 and 18 mole percent Y_2O_3 . Melts were prepared from the following raw materials.

- (1) Al_2O_3 - Linde A (0.3 μ) Crystal Products, Linde Division, Union Carbide Corporation
- (2) Y_2O_3 - Code 1116 Trona Chemicals, Rare Earth Division, American Potash and Chemical Corporation

The eutectic was mixed with de-ionized water in an alumina (~94% Al_2O_3) motor driven mortar and pestle (1/2 hour mixing time). This approach was used only to prepare preliminary small batches (<50 grams). Ball milling in a high alumina mill (99+% Al_2O_3) was the basic method used to prepare larger batches.

After mixing, the slurry was poured into a graphite crucible and dried in an air oven at 150°C for one hour.

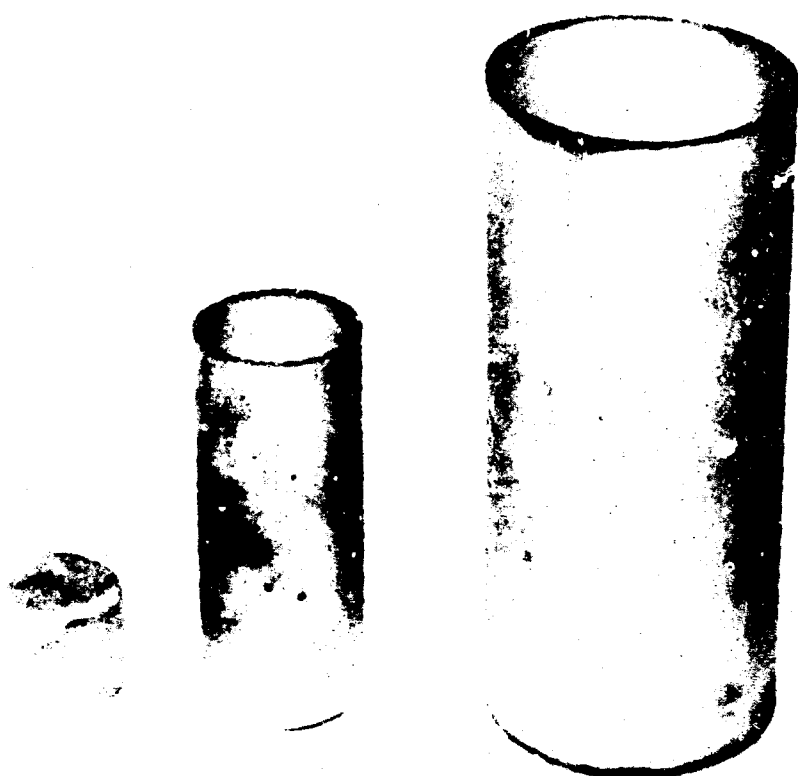


Figure 51. Crucibles Used in Aluminum Oxychloride Preparation

Several initial experiments (<30 grams) with induction heating in a flowing argon atmosphere demonstrated that the material could be melted satisfactorily at a temperature of approximately 1870°C (measured with an optical pyrometer). Each melted button was dark grey and contained numerous trapped gas pockets.

Approximately 30 grams of raw materials were melted in a graphite container in the oil diffusion pumped cold wall vacuum furnace. The furnace temperature was increased to 1870°C and held at this temperature for 1/2 hour at a pressure of approximately 1×10^{-4} torr. On cooling, it was apparent that the material had melted and formed a mass of rather large crystallites. A photomicrograph of the button surface is shown in figure 52 and photograph of the entire disc is shown in figure 53. The vacuum melted material shows a significant improvement in color and overall microstructure (no visible voids or gas pockets) over the one that was melted in argon using induction heating.

2. Ceramic and Ceramic/Metal Fabrications

This section describes specific materials and material combinations that were fabricated by the general methods previously outlined.

a. Ceramic Bodies by Hot Pressing

Five different oxide bodies were hot pressed in graphite dies after the powders were pre-formed or cold pressed in hardened steel dies. The hot pressing time-temperature relationship was varied to some extent for the different materials at a constant pressure of about 5000 psi. The major objective in this series of exploratory runs was to achieve high density with a minimum of experimental trials. Specific bodies are discussed below and table XXI summarizes the process data.

(1) Strontium Zirconates

Hot-pressed strontium zirconate (SrZrO_3) bodies were prepared from two grades of powder. Chemically pure (cp) grade strontium zirconate powder was supplied by Titanium Alloy Manufacturing Division, National Lead Company. The manufacturer reported the total impurity content to be less than 0.2%. Muscle Shoals Electrochemical Corporation supplied fused strontium zirconate powder. This powder was ground from an electrofused charge. No infor-

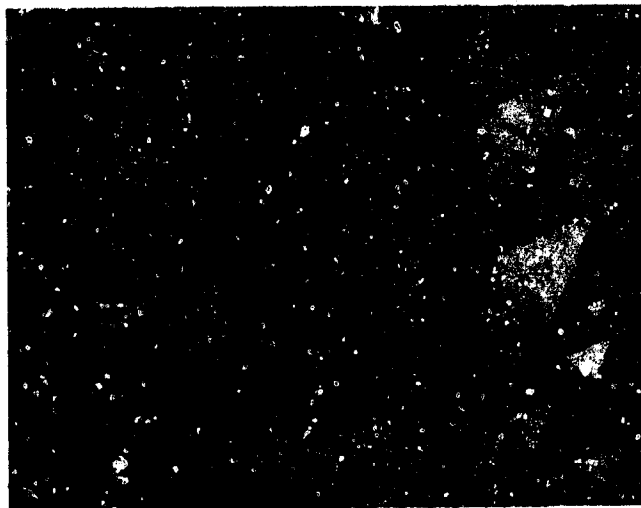


Figure 52. Vacuum Melted Alumina-Yttria
Microstructure

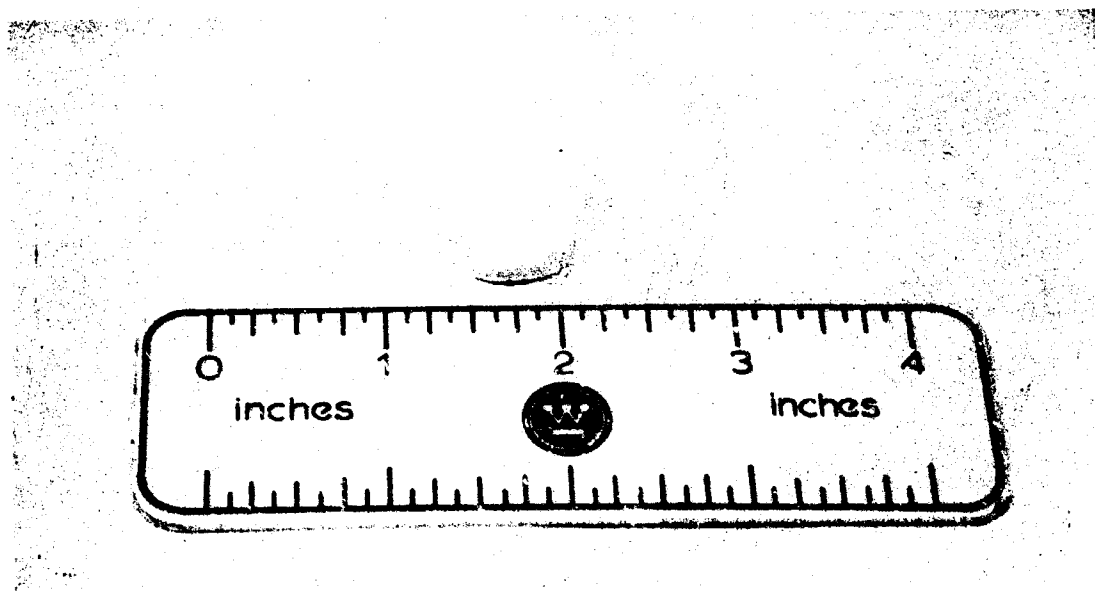


Figure 53. Vacuum Melted Alumina-Yttria Disc

TABLE XXI. Summary of Processing Data for Hot Pressed Ceramic Bodies

Body	Composition of Starting Material	Purity of Starting Materials	Hot Pressing Data	Comments
Al ₂ O ₃ /Y ₂ O ₃ K1935130	92 Mole % Al ₂ O ₃ 18 Mole % Y ₂ O ₃ wet milled	99.9%(Linde A) 99.99% (Y ₂ O ₃)	5000 psi for 83 minutes at 2600° to 2800°F	
SrZrO ₃ (fused) K1935128	Made from pre- fused and re- ground SrZrO ₃ powder	Unknown	5000 psi for 120 minutes at 2760° to 2850°F	Used Grafoil to prevent sticking to punches
SrZrO ₃ CP K1935127	Made from CP Calcined Powder	99.8%	5000 psi for 60 minutes at 2800°F	Used Grafoil to prevent sticking to punches
Y ₂ O ₃ K1935131	"As received" 2.10 micron powder	99.99%	5000 psi for 60 minutes at 2600° to 2800°F	
Sm ₂ O ₃	"As received" powder	99.9%	5000 psi for 35 minutes at 2600° to 2700°F	
Gd ₂ O ₃	"As received" powder	99.9%	5000 psi for 30 minutes at 2600°F	

mation was available on its purity; however, some contamination may have been introduced during the grinding and milling operations in addition to the impurities contained in the starting materials.

(2) Alumina-Yttria

A mixture of aluminum and yttrium oxides in the ratio of 82 mole percent Al₂O₃ and 18 mole percent Y₂O₃ was prepared by wet milling from the following materials:

- a) Al₂O₃ - Linde A, Crystal Products Department, Line Division, Union Carbide Corp.
- b) Y₂O₃ - Code 1115, American Potash and Chemical Corp., Rare Earth Division.

Both of these materials were very pure (>99.9%).
An analysis of Y_2O_3 is shown in table XXII.

The powdered materials were wet milled for 2 to 3 hours in a 99.7% alumina ball mill. After milling, the slurry was oven dried and then calcined in 99.7% alumina crucibles at 1300°C for one hour before pressing.

TABLE XXII. Analysis of Yttrium Oxide Used in Hot Pressing

Material: Yttrium Oxide Code 1116 Purity based on rare earth content Lot 130-81-68	
Lanthanum Oxide	<0.0019%
Cerium Oxide	<0.010
Praseodymium Oxide	<0.0039
Neodymium Oxide	<0.0019
Europium Oxide	<0.0015
Terbium Oxide	<0.020
Dysprosium Oxide	0.010
Holmium Oxide	<0.001
Erbium Oxide	<0.001
Thulium Oxide	<0.0005
Ytterbium Oxide	<0.00125
Lutetium Oxide	<0.0019
All other rare earths less than spectrographic limits	
Calcium	<0.0030
Silicon	<0.0035
Loss on ignition	0.33%
Particle size by Fisher Sub Sieve Sizer	2.10 microns

(3) Other Materials

Three other oxide materials were hot-pressed from "as received" powder supplied by American Potash and Chemical Corporation. These include:

Y_2O_3 - Code 1116 (Analysis - table XXII)

Sm_2O_3 - Code 823 (Analysis - table XXIII)

Gd_2O_3 - Code 929.9 (Analysis - table XXIII)

(4) Properties of Hot-Pressed Materials

Hot-pressed ceramic materials which were made at Westinghouse were checked for density and microstructure. Specimens were weighed in air and liquid (carbon tetrachloride). A correction was made for the weight of the platinum wire (specimen holder) suspended in the liquid. Densities were calculated from the following relationship:

$$\text{Density (gm/cm}^3\text{)} = \frac{\text{weight in air}}{\frac{\text{weight in air} - \text{weight in liquid}}{\text{density of liquid}}}$$

These densities are shown in table XXIV. Calculated densities are compared to theoretical values except for the alumina-yttria material. The accuracy of the measuring technique was checked by running several samples of Linde sapphire. Calculated densities were 3.98 and 3.96 gm/cm³ compared to a theoretical value of 3.98 ± 0.02 for hexagonal alumina.

Figures 20 through 26 show microstructures of all hot pressed ceramic materials. Listed below are estimated grain sizes of those prepared at Westinghouse.

- a) Y_2O_3 - 1 to 3 microns
- b) Chemically pure $SrZrO_3$ - 7 to 9 microns
- c) Pre-fused $SrZrO_3$ - 6 to 25 microns
- d) Al_2O_3/Y_2O_3 - 2 phases

TABLE XXIII. Analyses of Gadolinium and Samarium Oxides Used in Hot Pressing

Material: Gd_2O_3 Code 929.9	
Lot LX0610	
Purity based on rare earth content	
Lanthanum Oxide	0.0051%
Samarium Oxide	0.009%
Europium Oxide	<0.009%
Yttrium Oxide	0.012%
Material: Sm_2O_3 Code 823	
Lot LW0323	
Purity based on rare earth content	
Yttrium Oxide	(a)
Europium Oxide	0.034%
Gadolinium Oxide	<0.021%
Necdymium Oxide	(a)
(a) Not determined.	

All materials had a crystalline structure except the Al_2O_3 - Y_2O_3 eutectic composition which appears to be a two-phase system. One phase had a high population of residual polishing scratches (see figure 24) indicating that it was softer than the other phase.

b. Ceramic/Metal Hot Pressed Composites

Two alumina-rhodium end-to-end type composite discs were hot pressed at approximately 2550°F for 35 minutes. One disc was prepared with no cermet interface layer between pure rhodium end pieces. These rhodium end discs showed no bond to the alumina after removal from the die. The second disc contained a cermet (50v/o Rh-50v/o Al_2O_3) transition layer between the pure alumina

TABLE XXIV. Densities of Hot-Pressed Ceramic Specimens

Material (Process Number)	Sample Weight		Calculated Density (gm/cm ³)	Theoretical Density (2) (gm/cm ³)	Percent of Theoretical (%)
	Air (grams)	Liquid (1) (grams)			
Y ₂ O ₃ (K1935131)	0.4831	0.3305	5.02	5.03 (Cubic)	99.8
Gd ₂ O ₃ (K 1935133)	1.7238	1.3613	7.52	7.63 + 0.01 (Cubic)	98.6
Sm ₂ O ₃ (K 1935132)	2.5512	2.0215	7.62	7.62 (Cubic)	99.9
SrZrO ₃ - From Chemically Pure Powder (K 1935128)	1.4684	1.0381	5.43	5.48 (Cubic)	99.2
SrZrO ₃ - From Pre-Fused Powder (K 1935128)	1.3779	0.9621	5.26	5.48 (Cubic)	95.7
Al ₂ O ₃ / Y ₂ O ₃ K 19135135)	1.3277	0.8256	4.23	Unknown	--
Al ₂ O ₃ (Linde Sapphire)	0.5791	0.3487	3.98	3.98 + 0.02 (Hexagonal)	100
Al ₂ O ₃ (Linde Sapphire)	3.5574	2.1382	3.96	3.98 + 0.02 (Hexagonal)	100
(1) Carbon Tetrachloride, Fisher Reagent, Density (gm/cm ³) 1.584 ± .001 @ 25°C					
(2) J. F. Lynch et al (ED.), Engineering Properties of Selected Ceramic Materials, Journal of the American Ceramic Society, 1966.					

center discs and the rhodium end pieces. After hot pressing the pure rhodium end pieces were not bonded to the cermet. However, the cermet to alumina bond appeared to be quite good. Figure 54 shows the dimensions in cross section of the final configuration after a center hole was ultrasonically drilled. Figure 54 also shows a photograph of the specimen. The electrical resistance across each cermet face was less than one ohm; thus, indicating that the rhodium phase was continuous.

Comparing the results obtained for these two specimens demonstrates the significance of differences in thermal expansion coefficients. The total expansion of rhodium and alumina are as follows:

Rh (25° to 1500°C) - 1.79%

Al₂O₃ (25° to 1500°C) - 1.37%

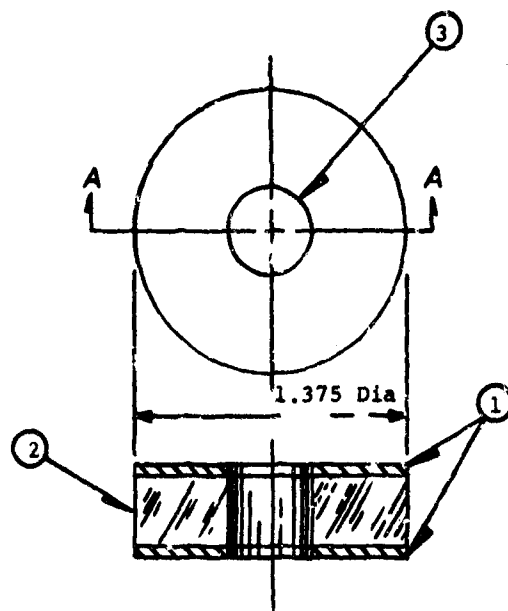
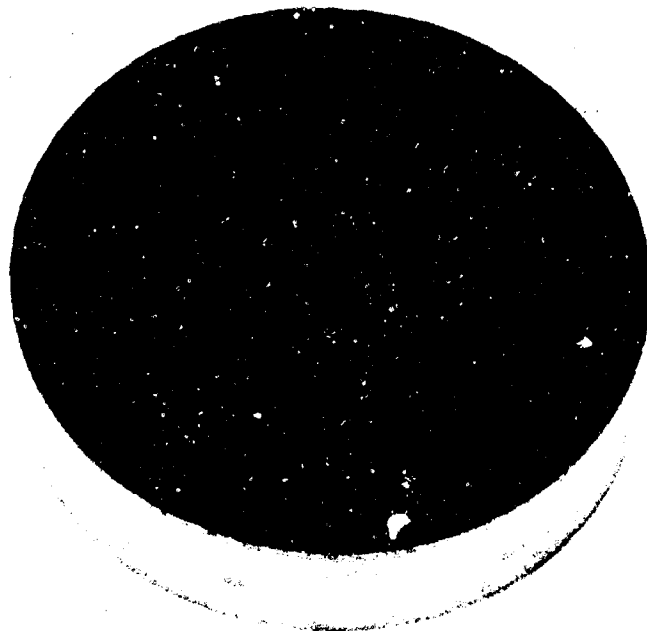
Rhodium expands about 31% more than alumina over the same temperature range. The stresses developed in the interface regions are apparently sufficient to rupture the rhodium/alumina bond as the composite is cooled to room temperature. To further minimize residual stresses and achieve a satisfactory metal to cermet bond additional graded cermet layers would be necessary.

c. Radio Frequency Sputtered Materials

Radio frequency (rf) sputtering was used in the application of both metallic and ceramic materials on various specimen substrates. Thin film metal deposits were used as binders between substrates and ceramic material. Ceramic thin films were either sputtered directly from ceramic sources or reactively from an aluminum source in an atmosphere containing oxygen. Some specimens were heat treated after sputtering in attempts to improve ceramic-substrate adhesion.

(1) Aluminum Oxide Films

Reactive diode sputtering of an aluminum target was investigated as a means of producing high purity Al₂O₃ films. A zone refined ultra high purity (>99.9%) aluminum target (Materials Research Corp.) was used in the diode configuration shown in figure 48. The reactive sputtering atmosphere was achieved by back-filling with pre-mixed argon and oxygen



- (1) 50 v/o Rh + 50 v/o Al_2O_3 - Approx. 0.015-Inch Thick
- (2) 100 v/o Al_2O_3 (Linda C) - Approx. 0.280-Inch Thick
- (3) 0.250 Inch Diameter Hole (ultrasonically cut)

Figure 54. Pressed Alumina-Rhodium/Alumina Composite

(50-50 v/o) gases to a pressure of about 5 microns. An insulating film approximately 2000 Å thick was deposited at a rate of about 35 Å/minute.

Lucalox (approximately 99.5% Al_2O_3 - 0.5% MgO) was sputtered directly on substrates in low pressure pure argon atmospheres. A deposition rate of about 100 Å/minute was obtained. Directly sputtered Lucalox was the predominant sputtered ceramic because of its higher deposition rate. The Lucalox sputtering conditions, were as follows:

Target and configuration

Horizontal

Diode configuration

One inch target-to-substrate distance

Lucalox target, 5-inch diameter by 1/16-inch thick with 4-inch diameter sputtered titanium electrode on back side.

Sputtering Conditions:

100% Argon at 5 microns pressure

Standing wave ratio - 1.5

Plate Current - 425 ma

Magnet Current - 6 amps

Sputtering time - 105 minutes

Deposition Rate - 100 to 120 Å/minute

(2) Titanium and Zirconium Films

Titanium and zirconium films were rf sputtered in argon using the diode configuration. Sputtering conditions were essentially the same as those outlined for Al_2O_3 except that zone refined, ultra high purity (>99.9%) metal target plates (Materials Research Corporation) were used. Deposition rates of about 80 Å/minute were obtained.

(3) Sputtered Specimens

Specimens were prepared using a variety of substrate materials and sputtered coatings. Most substrates were approximately 1/4-inch wide by 1-inch long. The following lists the types of substrates and coatings prepared as specimens.

<u>Substrates</u>	<u>Film and Thickness</u>
1) Niobium Foil, 0.001-inch thick:	Sputtered Lucalox ~12,000 Å thick.
2) Vanadium Foil, 0.001-inch thick:	Sputtered Lucalox ~13,000 Å thick.
3) Vanadium Foil, 0.001-inch thick:	Sputtered Lucalox ~24,000 Å thick.
4) Nickel Foil, 0.001-inch thick:	Sputtered Lucalox ~24,000 Å thick.
5) Tungsten Foil, 0.001-inch thick:	Sputtered Lucalox ~13,000 Å thick.
6) Tungsten Foil, 0.001-inch thick:	Sputtered Lucalox ~ 6,000 Å thick.
7) Molybdenum Foil, 0.001-inch thick:	Sputtered Lucalox ~13,000 Å thick. Post deposition heat treated in vacuum at 1800°F, 30 minutes (~3x10 ⁻⁵ torr).
The foil substrates listed above were all made from zone refined, ultra high purity stock (Materials Research Corp.).	
8) Hipercor 27 alloy 0.008-inch thick:	Sputtered titanium base layer 3000 Å thick. Sputtered Lucalox 13,000 Å thick. No post deposition heat treatment.
9) Hipercor 27 alloy 0.008-inch thick:	Same as 8) but re- ceived post deposition heat treatment in vacuum at 1800°F, 30 minutes (~3x10 ⁻⁵ torr).
10) One 4-inch Hipercor 27 Alloy Rowland Ring	Titanium base layer 3000 Å thick. Luca- lox ~ 13,000 Å thick.
11) Nickel-clad silver wire (transformer conductor material):	Sputtered Lucalox ~10,000 Å thick, over a length of about 3 inches. Ends of wire were plated over with nickel to pro- tect the silver core.

d. Magnetic Material Preparation and Insulation by Plasma-Arc Spraying

Hiperco 27 alloy was prepared and plasma-arc sprayed with alumina interlaminar insulation. This material was used as the magnetic material in both Rowland ring core box tests and the transformers, and was given short term potassium vapor exposure. Preparation of this material prior to plasma-arc spraying included punching or shearing, deburring, and various cleanings.

(1) Magnetic Material Preparation

Rowland rings and transformer E laminations were cut to size by punching and transformer I laminations by shearing. Both processes left burrs which were removed by the following chemical procedure:

- a) Etch in a solution of equal parts nitric acid, glacial acetic acid, and distilled water for one minute.
- b) Rinse in distilled water.
- c) Rinse in a 10% solution of sodium carbonate for five minutes.
- d) Rinse in distilled water for five minutes.
- e) Rinse in acetone for five minutes.
- f) Air dry.

Deburring left the Hiperco 27 alloy in need for further cleaning. This cleaning was performed according to the following:

- a) Dip in a solution containing equal parts of hydrochloric acid and distilled water.
- b) Rinse in distilled water.
- c) Rinse in chemically pure acetone.
- d) Dry with forced hot air.

Preparatory to annealing, laminations were dusted with magnesia to prevent sticking. Laminations were then wrapped in tantalum foil for added protection and annealed at 900°C for two hours in dry hydrogen. Following annealing, laminations were again cleaned (magnesia removed by wiping with cp acetone) and stored in a desiccator prior to the application of interlaminar insulation.

(2) Plasma-Arc Sprayed Ceramic Coatings

The main insulation material applied by plasma-arc spraying was high purity spherical alumina (99.9% Al_2O_3) obtained from Linde Division, Union Carbide Corporation. Spraying procedures for a uniform coating could not be established for the as-received material due to the wide range in particle size. Figure 55 shows the results of various screenings of this material. The material that was successfully applied as interlaminar insulation was that which would pass through a 325 mesh screen (-325 mesh = -44 microns = -0.0017 inch).

The following are the parameters used to apply an interlaminar insulation coating approximately 0.005-inch thick to Rowland rings, transformer E and I laminations, and several test specimens.

Chamber Atmosphere:

Argon at a temperature of 160° to 210°F and a pressure of approximately two inches water column above atmospheric
Impurities: Indicated oxygen less than 1 ppm. Indicated moisture 18 to 30 ppm.

Powder Feed:

Aspirator mode - Argon gas flow = 18 scfh at 40 psig.

Torch:

Argon flow = 100 scfh at 30 psig. Current = 450 amps d-c. Voltage = 38 volts d-c. Water pressure = 120 to 130 psig.

Torch Nozzle to Work Distance:

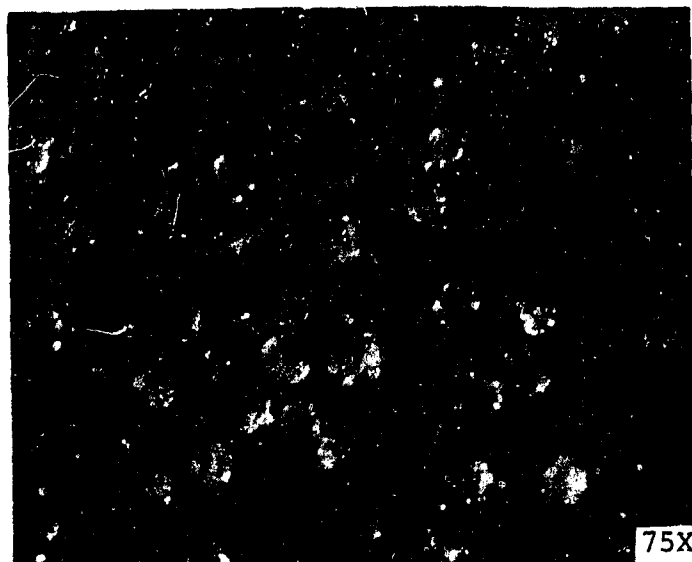
2-1/4 inches

Work Traverse Speed:

202 ipm

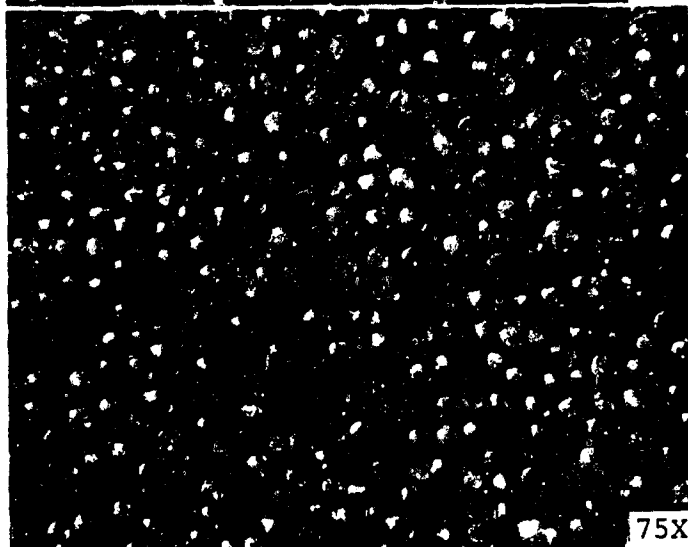
Torch Advance After Traverse Cycle:

1/8-inch

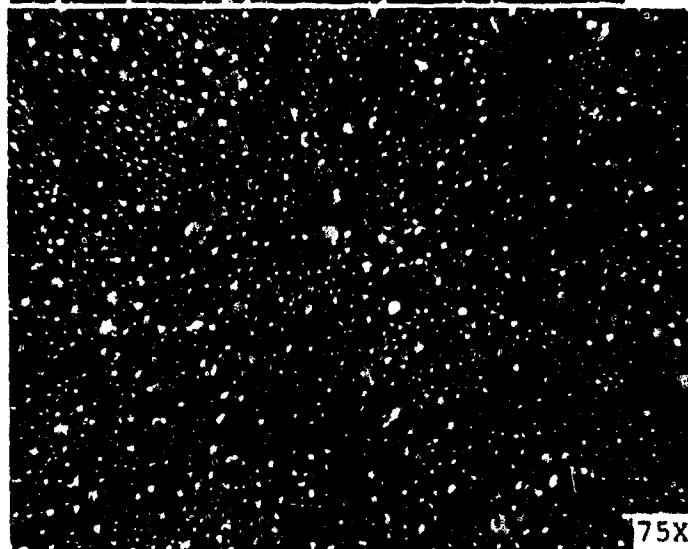


Screened Spherical
Alumina (99.9% Al_2O_3)

+ 200 Mesh
2%



-200 + 325 Mesh
19%



-325 Mesh
79%

Figure 55. Spherical Alumina After Grading

Figure 56 shows plasma-arc sprayed spherical alumina typical of that applied to transformer E and I laminations, Rowland rings, and short term potassium vapor exposure specimens.

e. Ceramic and Metal Tape Coils by Casting and Sintering

A flexible high-temperature potassium vapor resistant electrical conductor/insulation system was investigated. ("Flexible" refers to the ability to be flexed while in the green or unfired state.) Test samples consisted of 1/8-inch diameter rolled cores of yttrium oxide and rhodium composite tape approximately 3/16-inch wide. The tapes were cast as separate sheets (approximately 2 to 3 mils thick) on glass microscope slides using slurries of powered yttrium oxide or rhodium metal powder. Each slurry was made by mixing the proper powder with Fisher flexible collodion (nitrocellulose) in proportions that resulted in a thin molasses-like consistency. After air drying at room temperature, the sheets were separated from the glass, cut to size with a razor blade, and rolled together on a 1/8-inch diameter mandrel.



400X

Figure 56. Plasma-Arc Sprayed Spherical Alumina as Applied on Rowland Rings and Transformer E and I Laminations

Figure 57a shows a composite yttrium oxide-rhodium core (standing on end) and a core of yttrium oxide, all in the "flexible" green or unfired state. Figure 57b shows the same two coils after firing in air at 1150°C for one hour. Very little shrinkage or distortion occurred and the yttrium oxide was still very porous and weak. Figure 57c shows the same coils after vacuum firing (7.8×10^{-5} to 8.2×10^{-6} torr) at 1650°C for 5 hours. Some additional shrinkage occurred. The yttrium oxide became stronger but still showed considerable porosity. This approach to a conductor/insulation system appears promising; however, much more work would be required to produce a practical conductor/insulation system.

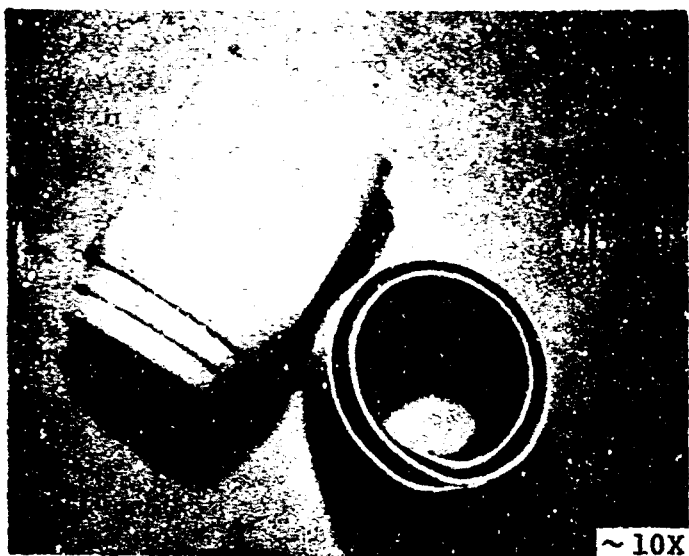
f. Application of Ceramic Materials by Other Methods

The application of potassium vapor resistant interlaminar and conductor insulations by processes other than plasma-arc spraying and sputtering were investigated. These processes were: bonding alumina by chemical reaction, depositing alumina by chemical decomposition, and bonding an eutectic mixture by melting it.

(1) Alumina Interlaminar Insulation

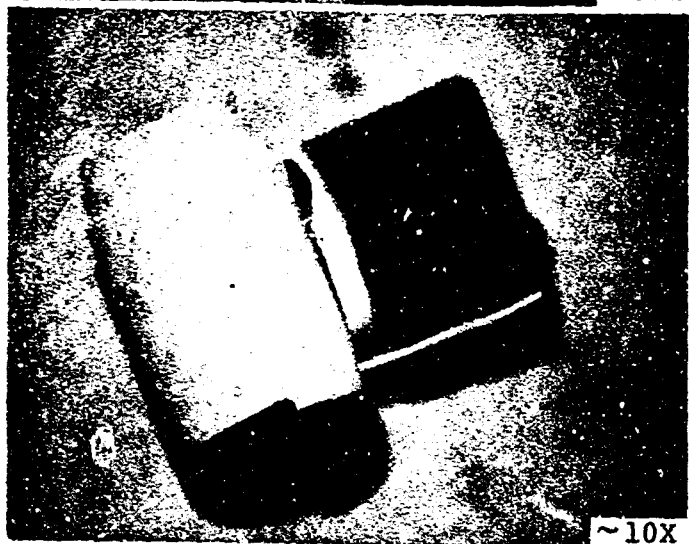
An interlaminar insulation investigation was conducted based on binding high purity alumina to Hiperco 27 alloy substrates by reaction with titanium from titanium hydride. The substrates for this investigation were Hiperco 27 alloy transformer E laminations either with or without nickel plating. These substrates were chemically cleaned with acetone prior to the application of the titanium hydride. The titanium hydride was in the form of -2 micron particle size powder that was mixed with a 10% amyl acetate-methyl cellulose solution. Paint and spray application techniques were investigated. Spraying with an air brush gave the most uniform coating.

The high purity alumina used was 0.05-micron size (Linde B) and 0.3-micron size (Linde A), both in 10% amyl acetate-methyl cellulose binders. Little difference could be detected in handling the two particle size powders. Brush application tended to remove the layer of titanium hydride thus air brush spraying was used to prepare specimens. Both the titanium hydride and alumina coatings were air dried at 200°F for one hour before further processing.



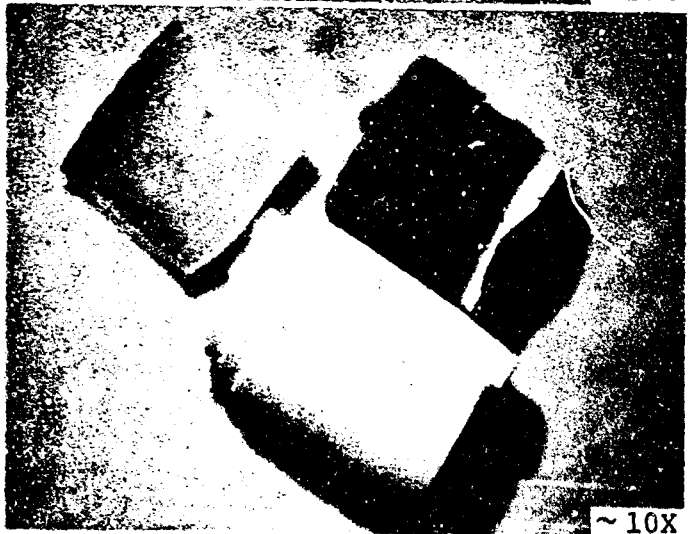
(a)

Photograph of
Unfired Coils



(b)

Photograph of
Coils After
Firing in Air
at 1550°C 1/2-
Hour



(c)

Photograph of
Coils After
Firing in Vacuum
at 1650°C, 5-Hours
(7.8×10^{-5} to 8.2×10^{-6}
torr)

Figure 57. Yttrium Oxide and Yttrium Oxide/Rhodium Tape Coils,
Fired and Unfired

Specimens both with and without the nickel plating were given one of the following heat treatments.

- a) 1000°C for 10 minutes in a hydrogen atmosphere furnace, cooled in nitrogen.
- b) 1000°C for 10 minutes in a nitrogen atmosphere furnace, cooled in nitrogen.
- c) 1000°C for 10 minutes in evacuated retort, cooled in vacuum.

In all cases the alumina failed to adhere to the substrates. A gray to black powder resulted with the lighter color being on the nickel plated substrates. The probable cause of the lack of adhesion is the incomplete decomposition of acetate-methyl cellulose binder.

(2) Alumina Conductor Insulation by Chemical Decomposition

Three-inch long specimens of 0.030-inch diameter rhodium and iridium wire were coated with aluminum oxychloride by dipping the coiled wire in an absolute ethyl alcohol slurry of aluminum oxychloride. Wires were resistance heated in air by passing a high alternating current at a low voltage through them. Wire temperature was monitored with an optical pyrometer. The rhodium wire was heated to temperatures high enough to melt rhodium, 1960°C³. During heating, the aluminum oxychloride coating underwent decomposition giving a densified alumina coating which appeared to have a sintered structure. The off-white aluminum oxychloride coating was observed visually to turn brown at approximately 400°C, then change to white at a higher temperature, and remain white after the temperature was lowered to room temperature (25°C). The coating was not well bonded to the rhodium and could be removed by scraping. Figure 58 shows rhodium wire with an alumina coating from the decomposition of aluminum oxychloride.

(3) Alumina-Yttria Conductor Insulation

Both pre-fused and mixed-calcined powders of alumina-yttria in the ratio of 82 mole % Al_2O_3 - 18 mole % Y_2O_3 were applied to rhodium conductor material. The

pre-fused material was a vacuum melted disc, which had been crushed in a diamond mortar and milled using a Pitchford Blender Mill with steel balls. The resulting average particle size was five microns. Both the crushing and the milling contaminated the material with steel. Steel was removed by boiling the powder in a 50% mixture of hydrochloric acid and distilled water and an attempt was made to filter out the powder. The powder went through the filter. Separation was made by rinsing and settling the powder and decanting off the liquid. After several rinses the powder was made into a slurry with absolute ethyl alcohol. The mixed-calcined powder was from the same material used in making hot-pressed discs as was described previously. This material was used in a slurry with distilled water.

The pre-fused and mixed-calcined powder were applied to rhodium wire from slurries. Brushing and dipping were used to apply the slurries with dipping giving the most uniform coating. Three coatings with an air dry between each coating were used to prepare the wires. Heating was accomplished in air by passing a high alternating current at a low voltage through the wire. At a temperature near the melting temperature of rhodium the pre-fused material melted and was drawn into droplets which adhered to the surface of the oxidized rhodium. Figure 59 shows the surface of the oxidized rhodium and fused droplets of adhering alumina-yttria. The mixed-calcined powder did not fuse or adhere when heated to the melting temperature of the rhodium wire.

3. Evaluation of Experimental and Commercially Available Materials in Potassium Vapor

Short term potassium vapor exposure tests were performed on specimens which were products from the previously described development or commercially available ceramic materials. Test durations were approximately 200 hours, and the test temperature was either 600° or 850°C.

a. Ceramics

Modulus-of-rupture tests were run on purchased and Westinghouse fabricated ceramic MOR bars both before and after short term potassium vapor tests. Table XXV gives statistical data on these tests and figure 60 shows absolute and relative MOR values.

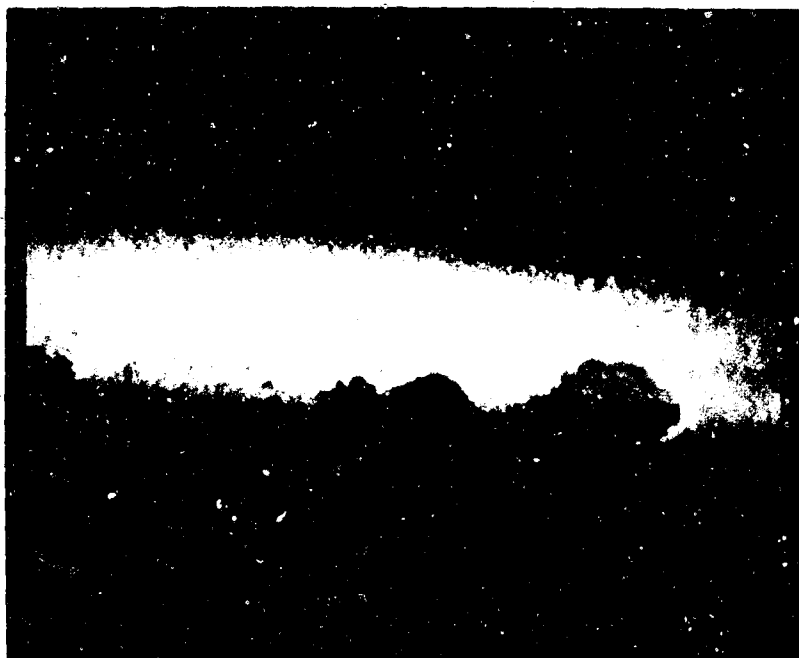


Figure 58. Alumina From Aluminum Oxychloride
on Rhodium Wire (40X)

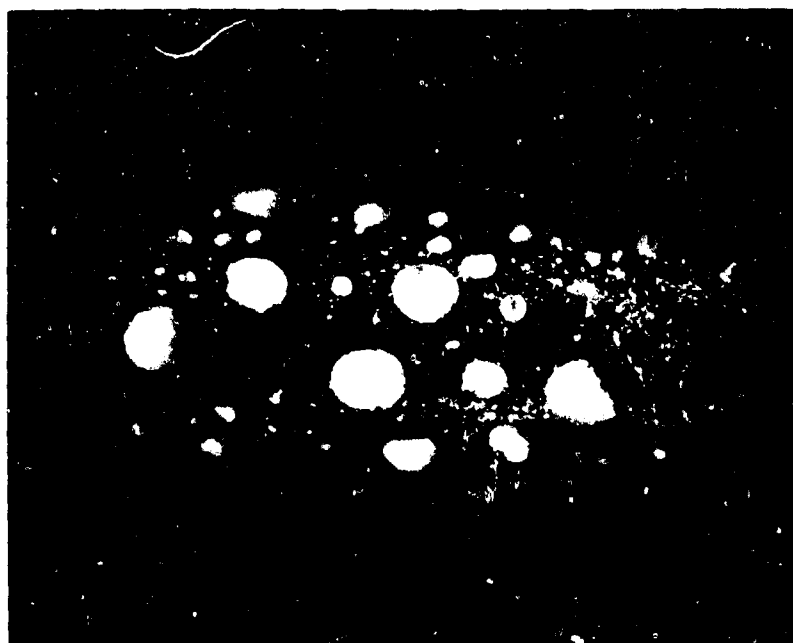


Figure 59. Alumina-Yttria From Pre-fused
Material on Rhodium Wire (40X)

TABLE XXV. Ceramic Materials and Modulus of Rupture Before and After Exposure to 850°C Potassium for about 200 Hours

Material	Composition	Processing	Average Modulus-of-Rupture					
			Before Exposure			After Exposure		
			MOR (psi)	Standard Deviation	Coefficient of Variation	MOR (psi)	Standard Deviation	Percent of Original MOR
SPINEL B	Proprietary (American Lava Corporation)	Pressed and Sintered	40,000 (2)*	2800	7.0%	33,133 (3)*	373	1.2%
SPINEL A	Proprietary (American Lava Corporation)	Pressed and Sintered	35,900 (2)*	3500	9.8%	6050 (2)*	Zero	16.9
ALUMINA 753	99.5 Al ₂ O ₃	Pressed and Sintered	40,525 (4)*	Not Calculated	--	All Specimens Broken After Unloading		
Alite A-610	U.S. Stoneware 99% Al ₂ O ₃ 1% MgO	Pressed and Sintered	35,850	2164	6.05	14,996	173	41.7
Alite A-612	U.S. Stoneware 97% Al ₂ O ₃ 3% SiO ₂	Pressed and Sintered	35,850	2164	6.05	Zero	--	Zero
ISOTROPIC CVD	~100% BN (Raytheon Co.)	Chemical Vapor Deposition	11,053	1874	16.9%	6,185	900	59
SrZrO ₃ Chemically Pure (CP)	Made from CP Grade Powder >99% SrZrO ₃	Hot Pressed at WLED	17,412	2000	11.5%	4,276	712	24.5
SrZrO ₃ Fused	Made from Fused and Re-ground powder	Hot Pressed at WLED	32,900	3440	10.4%	19,250	1890	58.4
Y ₂ O ₃	>99% Y ₂ O ₃	Hot Pressed at WLED	21,800	--	--	24,450	2980	100
Al ₂ O ₃ /Y ₂ O ₃	82 Mole % Al ₂ O ₃ 18 Mole % Y ₂ O ₃	Hot Pressed at WLED	53,600	4120	7.7%	50,600	1600	95
Gd ₂ O ₃	>99% Gd ₂ O ₃	Hot Pressed at WLED	25,950	6000	23.2%	18,264	2420	70
Sr ₂ O ₃	>99% Sr ₂ O ₃	Hot Pressed at WLED	11,183	4360	39.0%	3,072	416	27.5

* Numbers in parentheses equal amount of specimens

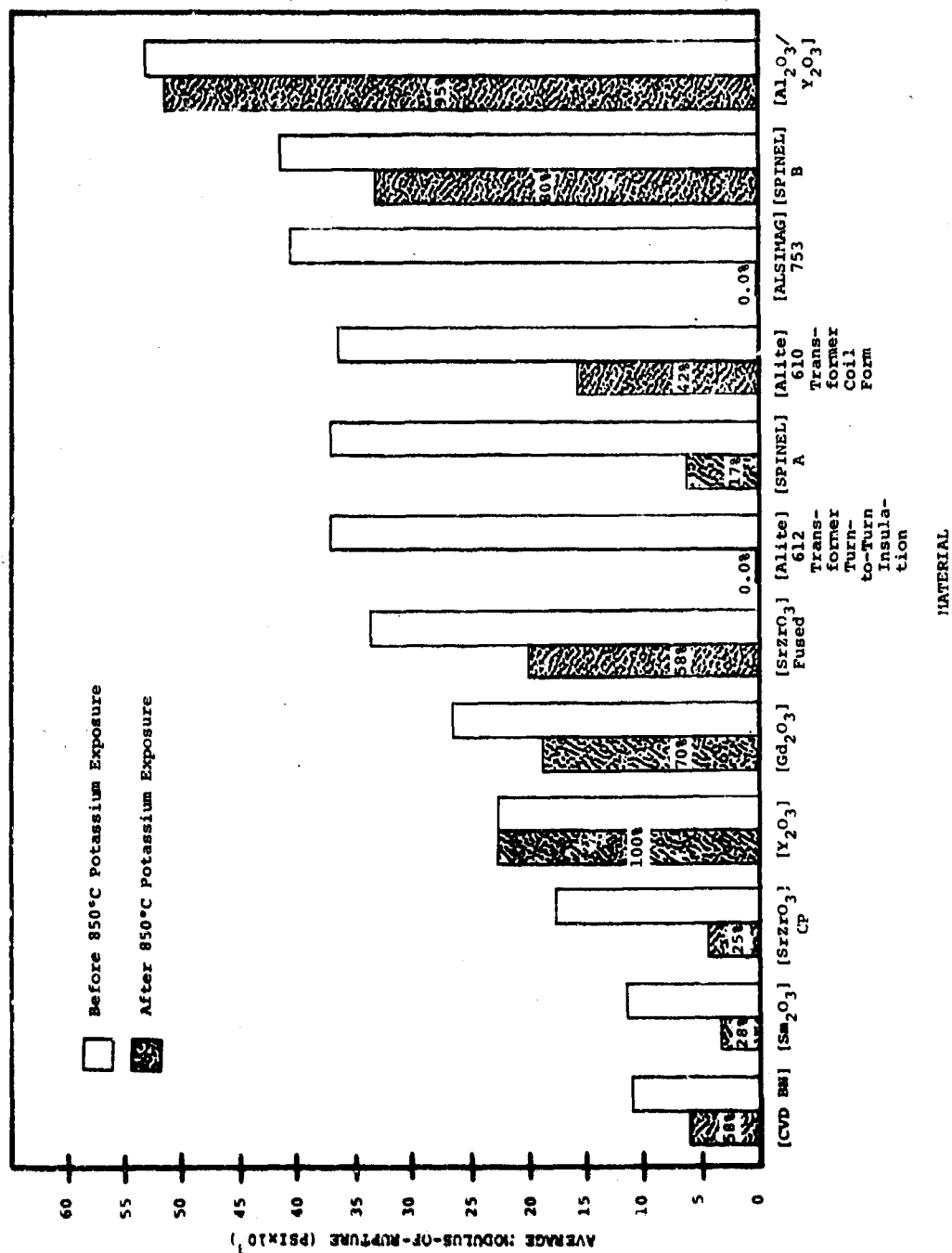


Figure 60. Comparison of Ceramic Materials (MOR bars) Before and After Exposure to 850°C Potassium Vapor for Approximately 200 Hours

These results are generally consistent with the longer term lower temperature tests where a comparison can be made between the same materials. For example, cp SrZrO_3 lost 75% of its original strength, fused SrZrO_3 lost 42% of its original strength, and Y_2O_3 showed no change in strength after 200 hours in 850°C potassium vapor. By comparison, these same materials lost 60%, 30%, and showed no change (respectively) after 5000 hours in 600°C potassium vapor. Hot pressed alumina-yttria eutectic composition ceramic showed high strength and low degradation consistent with that found in long term tests. The short term tests (850°C for 200 hours) appear to be at least as severe as the 5000-hour 600°C test.

Alite materials were also common to both long and short term potassium vapor exposure tests. Short term tests were run on specimens of the transformer coil form and turn-to-turn insulation materials. These tests were conducted to establish the potassium vapor resistance of the material after the 1000-hour test MOR bars lost all strength. Turn-to-turn spacers lost all strength and were later identified as Alite A-612, a 97% Al_2O_3 - 3% SiO_2 material, as were the 1000-hour test bars. Transformer coil form material, designated Alite A-610, had an after test MOR strength of 15,000 psi which is 42% of that average of other unexposed material designated as Alite A-610. After 2000-hours in 600°C potassium vapor, this other material retained 20% of its unexposed strength. The consistency of Alite A-610 material from batch to batch is questionable.

Two spinel structure ceramics from American Lava Division, 3M Company, were given short term tests. These materials, of proprietary composition, were known to contain silica, but were tested to obtain specific information on this potentially useful ceramic structure. These spinel structures do not appear to be sufficiently stable to prevent potassium from reacting with certain contained materials, thus reducing the strength of the material. The material designated spinel B showed a 20% decrease in MOR strength and remained stronger than all but one other material in this test series. Spinel A lost nearly 80% of its MOR strength.

ALSIMAG 753 was tested to evaluate a readily obtainable high alumina ceramic which would be available in many forms for use in potassium exposure test instrumentation.

This material initially appeared promising because of the metalizing and brazing technology which has been developed for its use in high temperature electrical applications. ALSIMAG 753 appears to contain materials which are not resistant to potassium as evidenced by the severe degradation from this test (see figure 60).

Rare earth oxides have relatively high negative free energies and thus in pure form should prove to resist potassium vapor. Hot pressed gadolinium oxide MOR bars showed a 30% decrease in strength and the samarium oxide showed a 72% decrease. The hygroscopic nature of these materials may be responsible for some or all strength degradations. A powdery surface layer was observed on samarium oxide MOR bars after they were sliced and cleaned. Pieces of this material lost all strength after aging in air for several months. A slight surface attack was also noted on specimens of gadolinium oxide after aging in air. Distilled water was used to remove potassium metholate during the potassium unloading process and may be at least partially responsible for the strength decrease in the specimens tested.

Raytheon Company's isotropic chemically-vapor-deposited (CVD) boron nitride is a ceramic which is relatively easy to machine and was investigated for use as an electrical insulation. This material lost over 40% of its strength during the exposure. Boron nitride of higher purity or prepared by other techniques may prove to be more stable in potassium vapor.

A specimen of Union Carbide Corp's yttria stabilized zirconia cloth was tested to help evaluate it for use as a transformer electrical insulation. A short term potassium vapor exposure test changed its color from nearly white before test to blue after test. This specimen was very friable after the test.

b. Ceramic/Metal Fabrication

Samples of promising ceramic/metal fabrications, prepared as described earlier, were tested to determine their electrical properties and their compatibility with potassium vapor.

(1) Electrical Tests Before Potassium Exposure

D-C electrical tests were run on Hiperco 27 alloy substrates coated with either plasma-arc sprayed or

sputtered alumina. Tests were run at 72°F and 68% relative humidity using a Keithly Electrometer (Model 610B) and a Keithly regulated d-c power supply (Model 240). This power supply has a circuit breaker that will trip at 10 ma.

Electric strength was measured on a Hiperco 27 alloy substrate with a 0.003-inch-thick coating of plasma-arc sprayed alumina. The thickness was determined after the test by examining the specimen's cross section using a calibrated reticle in a 30X microscope. Mercury was used as the counter-electrode and was confined to a 1.24-cm diameter by a plastic tube. The electrical breakdown occurred at 1110 V d-c.

Electrical strength of a typical 1.26-micron (4.96×10^{-5} inch) thick sputtered alumina coating was measured. A 1/8-inch diameter steel rod with flat ends was used against the coating as the positive electrode. Applied voltage was increased in increments as follows:

- a) 100 V d-c - held for 3 minutes
- b) 140 V d-c - held for 1 minute
- c) 190 V d-c - held for 1 minute
- d) 250 V d-c - shorted after 15 seconds

The electric strength of this film, based on the 250 V d-c, was 5000 V d-c/mil.

(2) Potassium Vapor Exposed Specimens

Substrates of Hiperco 27 alloy with plasma-arc sprayed (in air) coatings were exposed to 600°C potassium vapor for 200 hours then tested and evaluated. Titanium binder was applied to substrates from plasma-arc sprayed titanium hydride. Heat from the plasma decomposed the titanium hydride preventing oxidation of the deposited titanium layer. High purity alumina was plasma-arc sprayed over the titanium binder layer. Table XXVI gives coating thicknesses, processing, and results and evaluation after potassium exposure.

Visually, the insulating coatings appeared to be unaffected by potassium vapor exposure except for the samples that received the 2400°F vacuum heat treatment before exposure. Dark areas in these coatings were evident on several of these samples. Sample No. 2c was intentionally broken (Hiperco 27 alloy was quite brittle after exposure) to expose the cross section. Figure 61 shows a representation of the

TABLE XXVI. Process History and Observations of Hiperco 27 Alloy with Coatings Plasma-Arc Sprayed in Air After Approximately 200 Hours in 600° C Potassium Vapor

Process History				Observations and Test Data After Potassium Vapor Exposure (2)				
Sample No.	Coating Thickness (1)			Vacuum Heat Treatment (°F) (min)	Appearance (4) After 200 hrs in K Vapor at 600°C	Electrical Tests		
	Titanium (mils)	Alumina (Linde A) (mils)	(cm)			DC Resistance at 100 V d-c (72°F) ohms	Calculated Resistivity (ohm-cm)	DC Breakdown Voltage (3) (volts)
2c	2.5	3.0	7.6x10 ⁻³	2400 30	Gray with darker areas - adherence good	- - - - - (not measured)	- - - - -	- - - - -
2d	3.5	3.0	7.6x10 ⁻³	1800 30	Grayish coating	1.1x10 ⁸	1.8x10 ⁹	700
2i	2.0	1.0	2.54x10 ⁻³	2400 30	Speckled gray	4.0x10 ⁸	2.0x10 ¹⁰	200
1c	3.0	3.0	7.6x10 ⁻³	2400 30	Gray with darker area at one edge	2.0x10 ⁸	3.3x10 ⁹	1000
1d	3.0	3.0	7.6x10 ⁻³	1800 30	Grayish Coating	9.1x10 ⁸	1.5x10 ¹⁰	600
1i	2.0	1.0	2.5x10 ⁻³	2400 30	Speckled gray	Breakdown (5) before test voltage (100 V d-c)	--	50

(1) Plasma sprayed in air ambient on Hiperco 27. Substrate thickness measured with micrometer (peak to peak measurement).
(2) Keithly Regulated Power Supply (0-1000 volts), Model 240 and Model 610B Electrometer, Mercury counter-electrode (0.39 cm diameter).
(3) Voltage increased at 15 second intervals in 100 volt increments until power supply relay tripped (10 ma current flow).
(4) All 3 mil coatings appeared somewhat whiter before exposure to K vapor; adherence of coating on sample 2c was very good based on scratch test with a dental pick.
(5) Insulating coating too thin for electrical measurement.

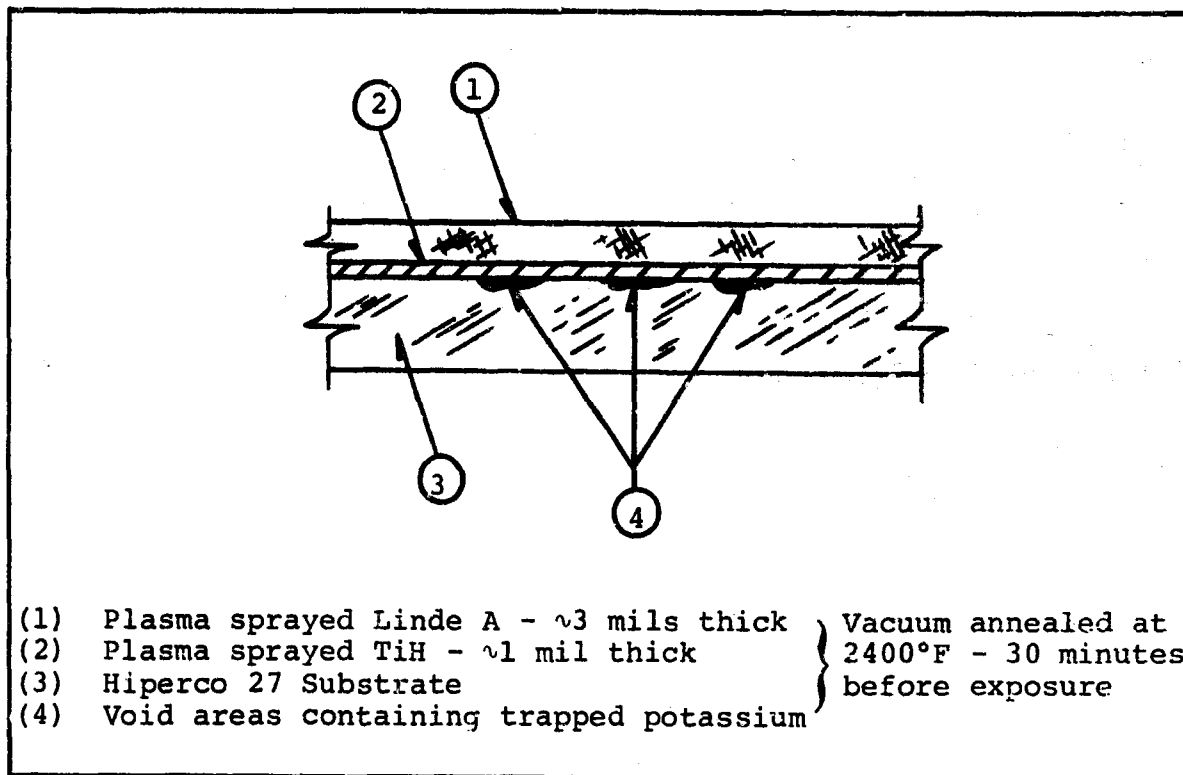


Figure 61. Cross Section of Plasma-Arc Sprayed Insulation on Hiperco 27 Alloy After 200 Hour Potassium Vapor Exposure at 600°C

observed interface regions. Void areas were visible at the titanium/Hiperco 27 alloy interface. An effervescence was observed within the void areas when the specimen was broken and freshly exposed surfaces were immediately inspected at 30X magnification. Evidently trapped potassium remained in these voids after the samples were removed from the test capsules and cleaned.

The electrical data shown in table XXVI provides additional evidence that the Linde A coatings exposed to 600°C potassium vapor have about the same d-c breakdown voltages (per mil of coating thickness) and d-c resistivities as unexposed specimens.

Hiperco 27 alloy substrates with plasma-arc sprayed coatings and/or sputtered coatings were exposed to potassium vapor at 850°C for 200 hours. Table XXVII describes the process history of these specimens and

TABLE XXVII. Process History and Observations of Hiperco 27 Alloy with Sputtered Coatings After 211 Hours in 850°C Potassium Vapor

Sample No.	Process History					Observations After Exposure
	Substrate	RF Sputtered Titanium Base Layer (MKC Zone Refined Target)	RF Sputtered Lucalox (99.5% Al_2O_3 - 0.5% MgO Target)	Coating Plasma-Arc Sprayed in Air	Vacuum Heat Treatment (3x10 ⁻⁵ Torr)	
1	Hiperco 27 Alloy 8 mils thick	~3000 Å Thick	~13,000 Å Thick	No Coating Applied	No Heat Treatment	90% of insulating coating remaining on substrate (see figure 62). Coating was cracked and buckled in some areas. Ohmmeter probing gave indications of a highly resistant surface.
7	Hiperco 27 Alloy 8 mils thick	~3000 Å Thick	~13,000 Å Thick	No Coating Applied	1800°F 30 min.	Insulating coating completely parted from substrate. Pieces of coating recovered from capsule (see figure 63). Low resistance surface. Substrate warped.
4	Hiperco 27 Alloy 8 mils thick	~3000 Å Thick	~13,000 Å Thick	Linde C ~2 mils thick	1800°C 30 min	Insulating coating completely parted from substrate. Pieces of coating recovered from capsule. Low resistance surface. Substrate warped.
9	Hiperco 27 Alloy 8 mils thick	~3000 Å Thick	~13,000 Å Thick	Linde C ~2 mils thick	No Heat Treatment	Insulating coating completely parted from substrate. Pieces of coating recovered from capsule. Low resistance surface. Substrate warped.
11	Hiperco 27 Alloy 8 mils thick	~3000 Å Thick	No coating	Alite 610 ~2 mils thick	No Heat Treatment	Insulating coating completely parted from substrate. Pieces of coating recovered from capsule. Low resistance surface. Substrate warped. Potassium leaked out of this capsule.
2	Hiperco 27 Alloy 8 mils thick	~3000 Å Thick	~13,000 Å Thick	Alite 610 ~2 mils thick	No Heat Treatment	Insulating coating completely parted from substrate. Pieces of coating recovered from capsule. Low resistance surface. Substrate warped.

the effects of potassium exposure (second 200-hour test). Additional details on the processing and electrical properties of these specimens were described previously.

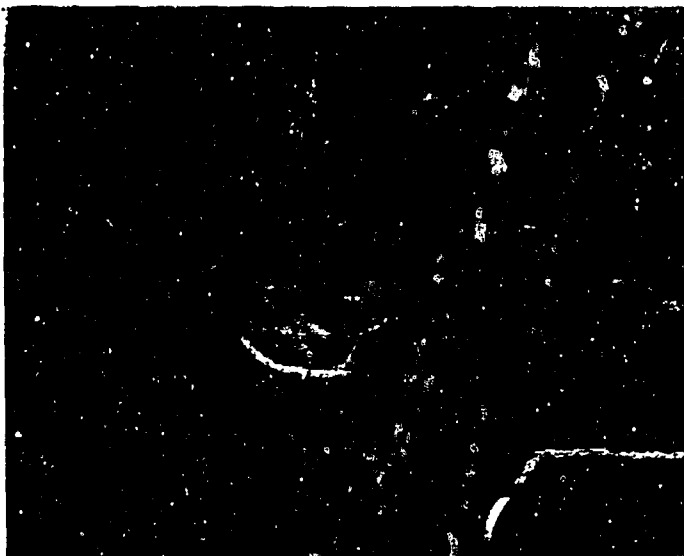
Sample No. 1 was the only sample that showed evidence that the insulating film was still attached to the Hiperco 27 alloy substrate after potassium exposure. The insulation of these specimens consists of a rf sputtered titanium base layer overcoated with rf sputtered Lucalox (99.5% Al_2O_3 , 0.5% MgO)

(~13,000 Å thick). These specimens received no post deposition vacuum heat treatment. Figure 62 is a photomicrograph of this coating after exposure to potassium. Cracks in the film are evident in this photograph. The coating appears to have contracted or buckled in some areas, thus exposing the surface of the Hiperco 27 alloy substrate, and remained flat and smooth with no evidence of cracks or peeling in others. The area shown in figure 62 is not typical of the entire surface of the film.

Figure 63 shows a photomicrograph, taken with transmitted light, of a piece of the sputtered Lucalox film that originally covered the Hiperco 27 alloy substrate on sample No. 7. This sample received a post deposition vacuum heat treatment (1800°C, 30 minute). None of the original coating could be detected on the substrate. Similar results were observed on the other sample groups that had plasma-arc sprayed coatings of high-purity alumina applied over base coatings of sputtered Lucalox, sputtered titanium, or on bare Hiperco 27 alloy.

These results indicate the following:

- a) Radio frequency sputtered Lucalox shows considerable promise as potassium resistant insulation on metallic surfaces. It appears that the sputtered Lucalox film is intrinsically resistant to potassium at 850°C. The buckling and cracking of the film on sample No. 1 might be attributed to stresses in the film resulting from thermal cycling materials with different thermal expansion rates.
- b) Sample Nos. 1 and 7 were prepared with the same type coatings but only sample No. 7



Sample No. 1 - R-F
Sputtered Lucalox coating on Hiperco 27 alloy substrate (sputtered titanium base layer - 1) after exposure to 850°C potassium for 211 hours. Photographed with reflected light at 400X.

400X

Figure 62. Sputtered Titanium and Lucalox on Hiperco 27 Alloy Substrate After Short Term 850°C Potassium Vapor Exposure (400X)



Sample No. 7 - R-F
Sputtered Lucalox (titanium base layer); originally attached to Hiperco 27 alloy substrate. Photographed with transmitted light at 200X.

200X

Figure 63. Sputtered Lucalox Film from a Hiperco 27 Alloy Substrate After Short Term 850°C Potassium Vapor Exposure (200X)

received a post deposition vacuum heat treatment. The sputtered coating in No. 7 came off the substrate after potassium exposure. A possible explanation for this is that the titanium base layer was oxidized during vacuum heat treatment and this oxide interface then dissolved in the hot potassium thus separating the Lucalox film from the substrate.

- c) Plasma-arc sprayed coatings on Hiperco 27 alloy with and without post deposition heat treatments and sputtered base layers did not remain adherent to substrates after 850°C potassium exposure.

Another group of sputtered specimens was prepared and exposed to 850°C potassium vapor for 200 hours. Specimen descriptions and observed results from this group are given in table XXVIII. Table XXVIII results are inconsistent with table XXVII results for sputtered Lucalox films on Hiperco 27 substrates. Sample Nos. 1 and 7 in table XXVII, and sample groups I and X in table XXVIII were respectively identical. Table XXVIII shows that group I specimens (vacuum heat treated 1800°F, 30 minutes) still had an insulating film after potassium exposure and group X specimens (not vacuum heat treated) showed no evidence of the original sputtered film on its surface. These observations are reversed for samples 1 and 7 in table XXVII. Figure 64 is a photograph of a table XXVIII group X specimen after potassium exposure. The darker areas of the surface is where the sputtered film was brittle and non-adherent. The film could be easily peeled from the surface indicating that interface corrosion may have occurred. The sputtered Lucalox film itself is relatively unaffected.

The other sample groups shown in table XXVIII include a number of one-mil thick foil substrates with sputtered Lucalox coatings (Niobium, Vanadium, Nickel, Tungsten and Molybdenum). From this group of specimens, the niobium, vanadium and tungsten substrates showed some evidence that at least part of their surfaces were still covered with the original sputtered Lucalox film. Figures 65 and 66 are photographs of the tungsten and vanadium specimens after exposure to potassium and show some areas still covered with an insulating film. Figure 66 (Vanadium) shows that the original sputtered film has broken up into flakes (possibly due to internal stresses).

TABLE XXVIII. Process History and Results of Sputtered Specimens After 200 Hours in 850°C Potassium Vapor

Sample Group	Substrate	Process History				Results After Exposure
		RF Sputtered Titanium Base Layer (MRC Zone refined target)	RF Sputtered Lucalox (99.75%Al ₂ O ₃ 0.25%MgO)	Plasma Sprayed Coating	Post Deposition Vacuum Heat Treatment (3x10 ⁻⁵ torr)	
IX	Niobium 1-mil-thick foil (MRC Zone re-refined)	none	13,000 Å	none	none	Insulating coating completely parted from substrate over 90% of surface. Small areas on surface were insulating, based on ohmmeter probing.
XI	Vanadium 1-mil-thick foil (MRC Zone refined)	none	1 sample 13,000 Å thick 1 sample 24,000 Å thick	none	none	13,000 Å sample - sputtered coating still on surface but highly crazed (see figure 66) 24,000 Å sample - no evidence of insulating coating on substrate.
VIII	Nickel 1-mil-thick foil (MRC Zone refined)	none	24,000 Å thick	none	none	No evidence (ohmmeter probe and visual inspection) of coating on substrate.
XII	Tungsten 1-mil-thick foil (MRC Zone refined)	none	1 sample 13,000 Å thick 1 sample 6000 Å thick	none	none	13,000 Å sample - most of surface was bare, small areas show visual (see figure 65) indications of insulating film. 6000 Å - no evidence of coating on substrate.
VI	Molybdenum 1-mil-thick foil (MRC Zone refined)	none	13,000 Å thick	none	1800°F 30 min.	No evidence of insulating coating on substrate.
I	Hiperco 27 alloy 8-mils-thick	3000 Å thick	13,000 Å thick	none	none	No evidence of insulating coating on substrate.
X	Hiperco 27 alloy 8-mils-thick	3000 Å thick	13,000 Å thick	none	1800°F 30 min.	Over 1/2 of surface shows evidence of insulating coating (see figure 64). Film could be easily parted from substrate but appeared coherent.
VII	Hiperco 27 alloy 8-mils-thick	none	none	Spherical alumina plasma-arc sprayed in argon atmosphere.	none	Alumina remained bonded to Hiperco 27 alloy substrate. No apparent evidence of corrosion.
II	Nickel-clad Silver. Wire ends plated with nickel.	none	10,000 Å over 3-inch length	none	none	No evidence of insulating film on conductor.
IV		Y ₂ O ₃ - Rhodium rolled coil (refer to section				Sample appeared to be unaffected by exposure (visual examination)
XIII		Alumina - Alumina Rhodium Composite (refer to section for details on fabrication)				Sample was unaffected by exposure (visual examination)



Figure 64. Hipercro 27 Alloy With RF Sputtered Lucalox Film
After Exposure to Potassium for 200 Hours at
850°C (16X)

Group VII consisted of three Hipercro 27 alloy specimens with a 0.2-mil-thick plasma-arc sprayed spherical sapphire coatings (applied in an argon atmosphere). Visual examinations showed that the coating was not lost or destroyed by potassium exposure. A nickel-clad silver wire specimen with a sputtered Lucalox coating, group II, showed no evidence of any remaining coating after exposure to potassium. In general, all of the sputtered film specimens show evidence that the original film adherence was destroyed after exposure to potassium and in some instances the structural integrity of the film was lost.

The group IV specimen was the composite yttrium oxide-rhodium coil described earlier. Although the coil was broken during removal from the test capsule there was no visual evidence of potassium attack. The rhodium conductor portion of the coil showed low resistance (<1 ohm) and overall electrical continuity. The yttrium oxide insulation appeared the same as it did prior to exposure. The alumina-alumina/rhodium hot pressed composite was designated group XIII. No evidence of degradation was found after exposure in either material or at the interfaces.

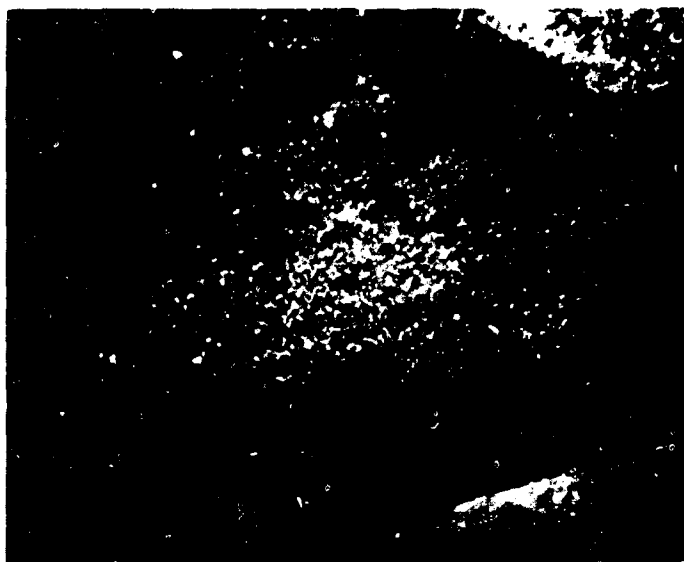


Figure 65. One-Mil-Thick Tungsten Foil with RF Sputtered Lucalox Film After Exposure to Potassium for 200 Hours at 850°C (16X)



Figure 66. One-Mil-Thick Vanadium Foil with RF Sputtered Lucalox Film After Exposure to Potassium for 200 Hours at 850°C (16X)

C. DESIGN FABRICATION AND EVALUATION OF 5-KVA TRANSFORMERS

Two 5-kVA transformers were built and tested. These devices were built using materials which were found suitable in a previous program⁵. Technologies for transformer fabrication and testing were advanced on this program. Transformers were tested both without and with potassium, and in some of the latter cases with the potassium intentionally ionized.

1. General Description and Design

Transformers for this program were of a conventional type⁸, but of potassium vapor resistant materials. Figure 67 gives a conceptual view of a transformer. Transformers were designed with proportionately more magnetic material weight than conductor material weight. This condition resulted in high iron loss and low conductor loss when the unit was operated at room temperature, and low iron loss and high conductor loss when the unit was operated at 600°C. The magnetic core cross sectional area was set near the value that would produce an optimum design based on the transformer rating, frequency, and flux density. The actual design deviated somewhat from the optimum by the selection of a standard lamination size so the laminations could be made on available tools.

The magnetic core was designed to operate at a flux density of 14.0 kilogauss at the nominal input voltage of 120 volts and frequency of 400 Hz. The core was 1.500 inches wide and 2.250 inches high with a 0.85 stacking factor. The conductors were designed to operate at approximately 2300 amperes per square inch with the transformer delivering full load current. The transformer had approximately 11 pounds of Hiperco 27 alloy and 4.5 pounds of 20% nickel-clad silver.

2. Fabrication of Transformer Components

Transformers consisted of laminated cores, primary and secondary conductors, and electrical insulation. Fabrication details for these components are given below.

a. Primary and Secondary Conductor Coils

Electrical conductors were 0.081-inch by 0.129-inch rectangular wire consisting of a silver core with 20% nickel cladding. Two of these conductors, electrically in parallel and physically located in a two-high arrangement were used in both primary and secondary windings. One transformer was assembled using separate pairs of conductors for each row (i.e., both primary and secondary windings consisted of joined rows). The conductor

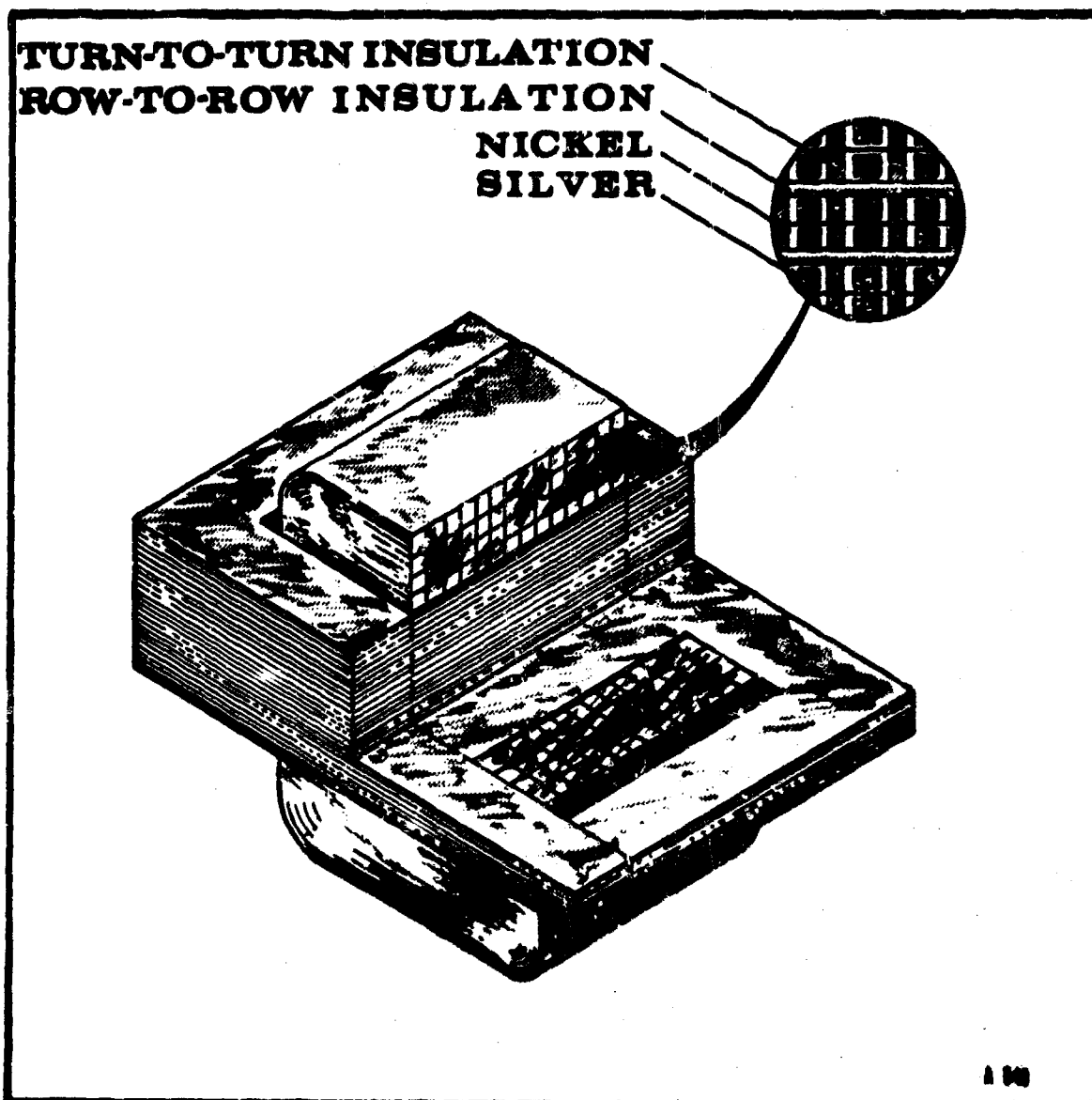


Figure 67. Conceptual View of 5-kVA Transformers

used here was the redrawn material which was only partially annealed as-received. This conductor was vacuum annealed: 800°C, for two hours at a pressure of 1×10^{-9} torr. Figure 68 shows conductors for the joined winding transformer. Each winding was formed around an appropriate mandrel.

The other transformer was wound with the two layer rows of each winding continuous. The inner rows were formed over mandrels shown in figure 69. The outer rows were formed over the inner rows with metal spacers (which made room for ceramic row-to-row spacers) located between. The conductor used in the continuous windings transformer was sufficiently annealed so that windings could be made with the material as received with no further annealing.

b. Laminated Core

The transformer cores were made of Hiperco 27 alloy from the same coil as that used elsewhere in the program. E laminations were punched and I laminations sheared to the dimensions shown in figure 70. Laminations were deburred, annealed, cleaned, and plasma-arc sprayed with alumina interlaminar insulation as was described previously.

c. Electrical Insulation

All transformer electrical insulation was ordered as low silica Alite A-610. The coil form, which fit around the center of the E laminations, was made to dimensions shown in figure 71. Transformer row-to-row insulation spacers were made according to figure 72. Turn-to-turn insulation spacers were 0.020-inch thick by 0.163-inch high and either 1.51 or 2.35 inches long. All Alite A-610 parts were diamond ground to size. Outside the outer row of the secondary of each transformer were plates of Alite A-610 fabricated as row-to-row spacers.

3. Transformer Assembly

Transformers were built from the previously described parts. These parts were either cleaned during previous processing or were cleaned in chemically pure acetone just prior to assembly. All parts were handled either with white gloves or with clean tools. General transformer assembly is given in the following section on the continuous winding transformer. Differences in assembly of the joined winding transformer are discussed in the second following section.

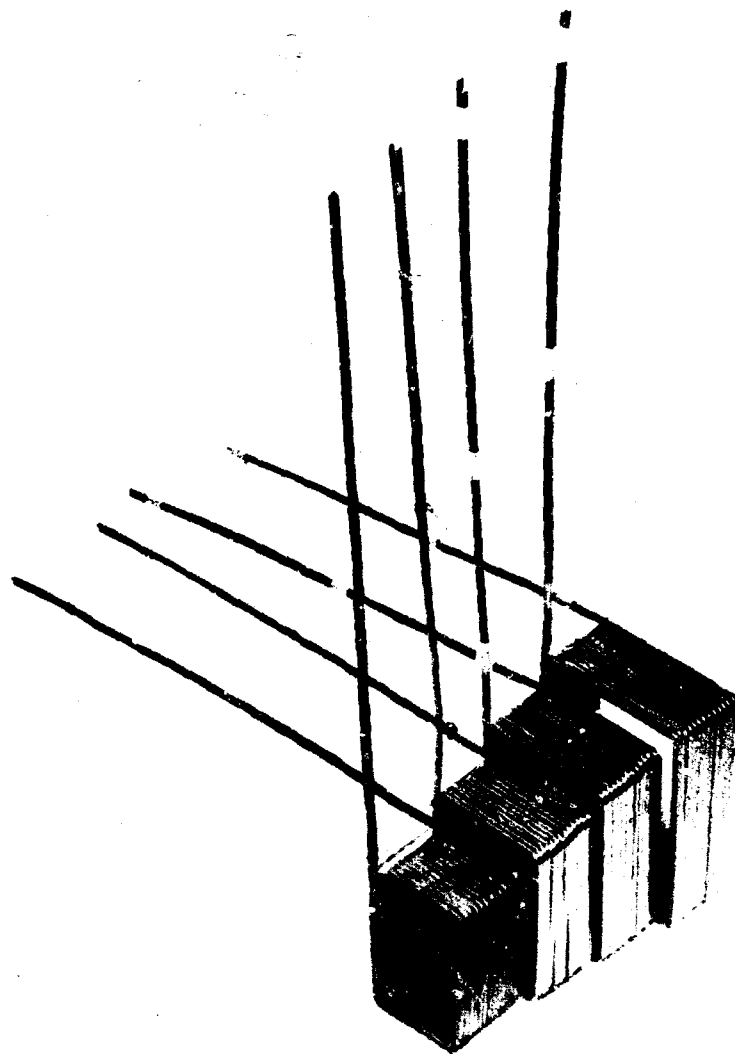


Figure 68. Transformer Windings - Joined Windings Transformer

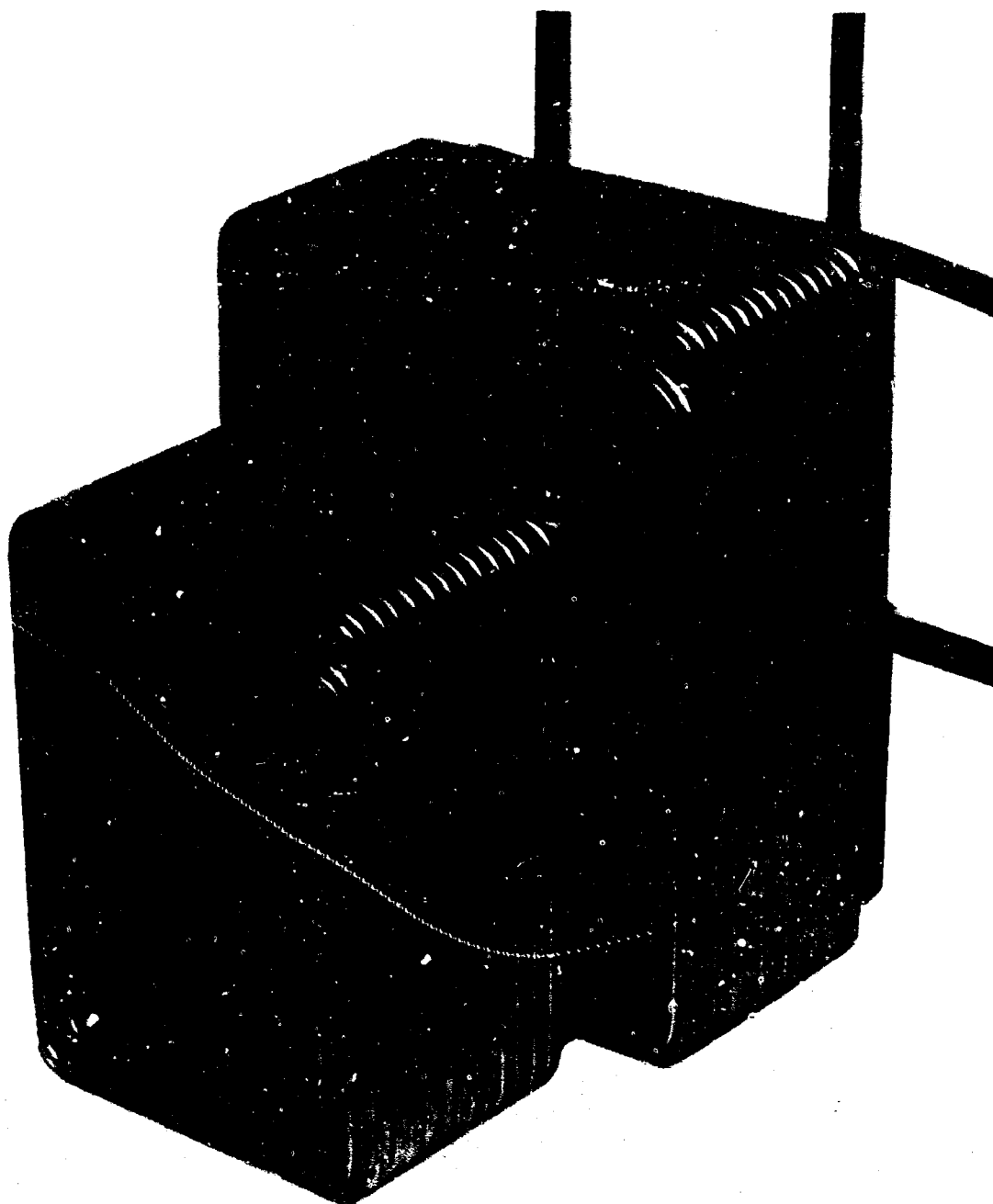
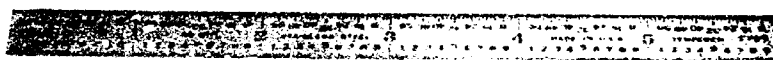


Figure 69. Transformer Windings - Continuous Windings
Transformer (Fixtures Shown)



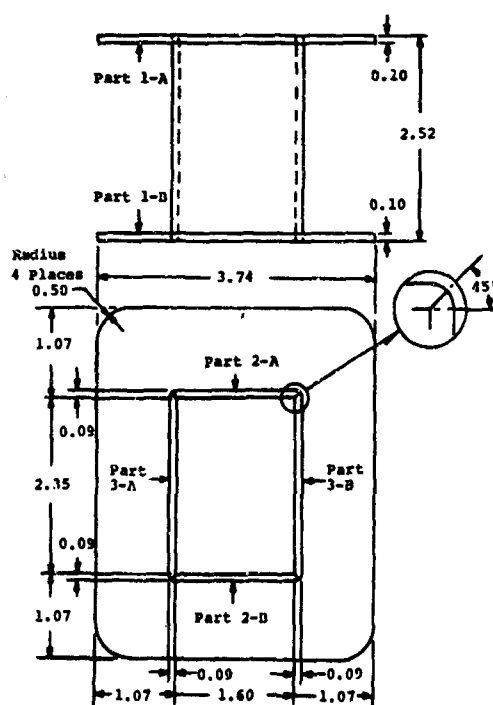
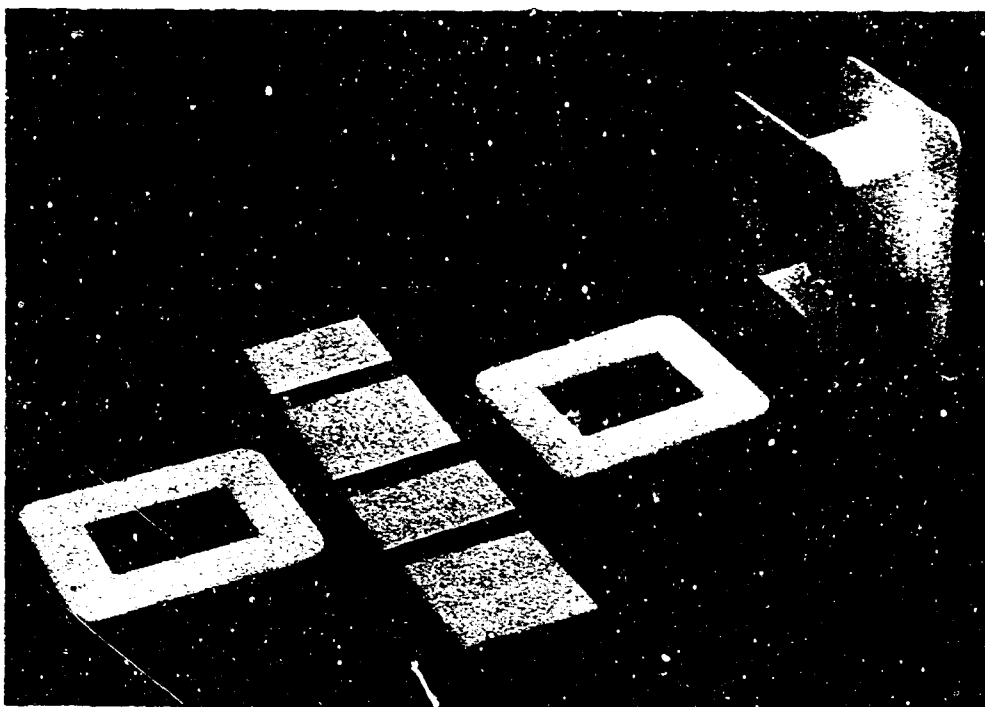


Figure 71. Transformer Coil Form

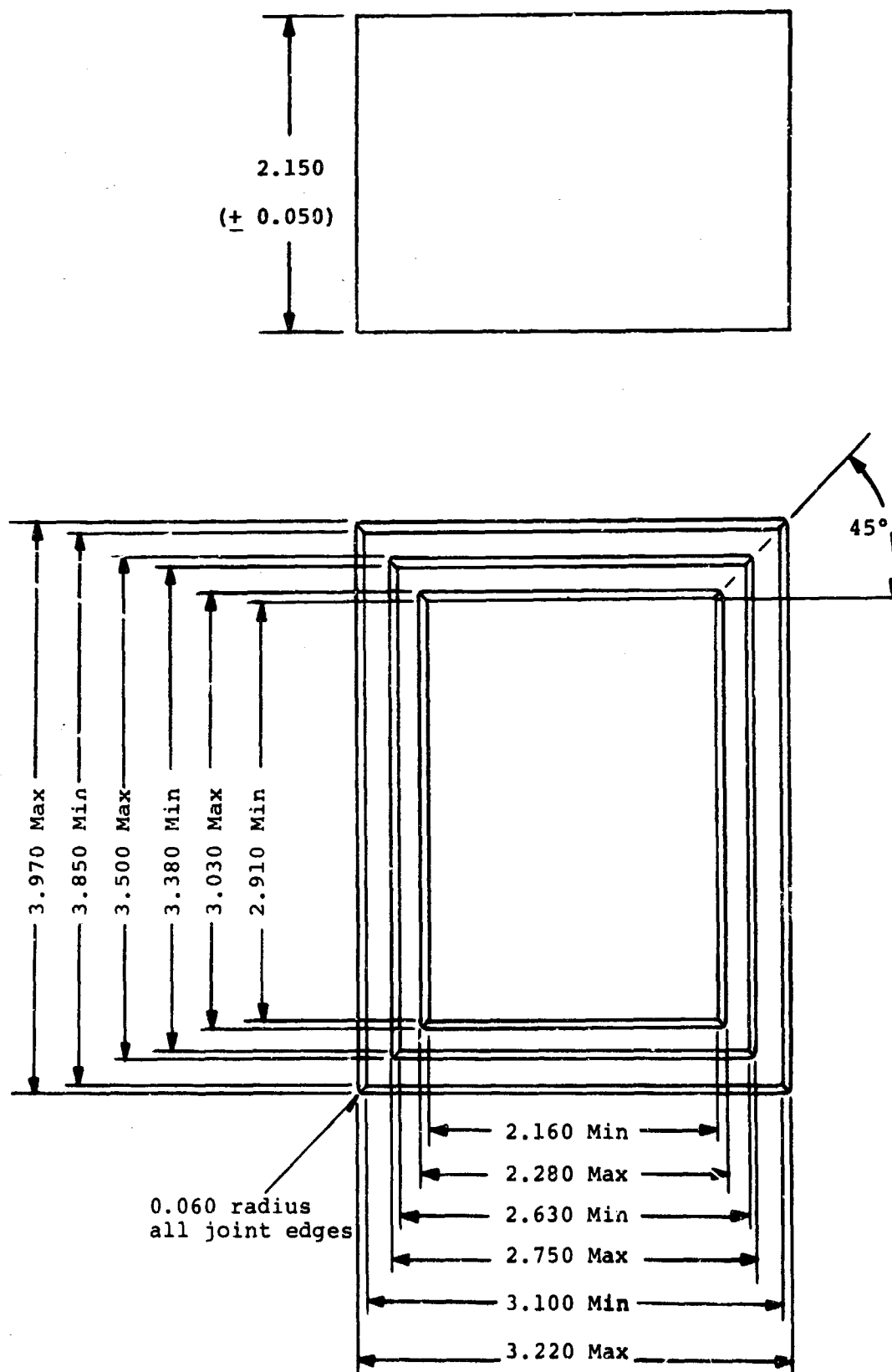


Figure 72. Transformer Row-to-Row Insulation

a. Continuous Windings Transformer

Transformer assembly began by insulating the conductor coils. Turn-to-turn insulation spacers were placed between coil turns and secured in place with either row-to-row insulation spacers or coil-form parts. The primary coil was then inserted physically inside the secondary coil and the transformer conductors assembly was secured as a unit by the addition of the coil form end plates. The transformer core was assembled around the conductors assembly by inserting E laminations alternately from each of two sides (insulated surfaces always against uninsulated) and placing an I with each E to complete the magnetic path. The transformer unit was completed by clamping laminations with stainless steel end plates which were bolted together. Figure 73 shows the assembled continuous winding transformer before leads were shaped, cut to length, and plated.

b. Joined Windings Transformer

The joined windings transformer was assembled in generally the same manner as the continuous winding transformer. One difference was that windings were completed by joining conductors from appropriate rows. Conductor nickel cladding was removed from conductor ends and conductors to be joined were secured to each other with exposed silver cores touching. Conductor joining was accomplished by fusing silver core material together using TIG welding in an inert atmosphere. The inert atmosphere was used to exclude oxygen which, if present, would be absorbed by the silver and possibly later rupture nickel plating. These joints were then etched and nickel plated to protect the silver from potassium attack.

A second difference between transformers was in the insulation where the primary (inside) winding crossed over the secondary (outside) winding. The joined winding transformer primary crossover insulation was 0.060-inch-thick plates; whereas, the continuous windings transformer had 0.020-inch-thick turn-to-turn spacers.

4. Design and Fabrication of Transformer Test Containers

Two transformer test containers were designed and fabricated of potassium vapor resistant materials. Test container functions were to fixture the transformer, contain the potassium test environment, and provide for electrical power and test instrumentation. The basic container parts of each were a 5.25-inch length of 8-inch schedule 40 seamless pipe and two

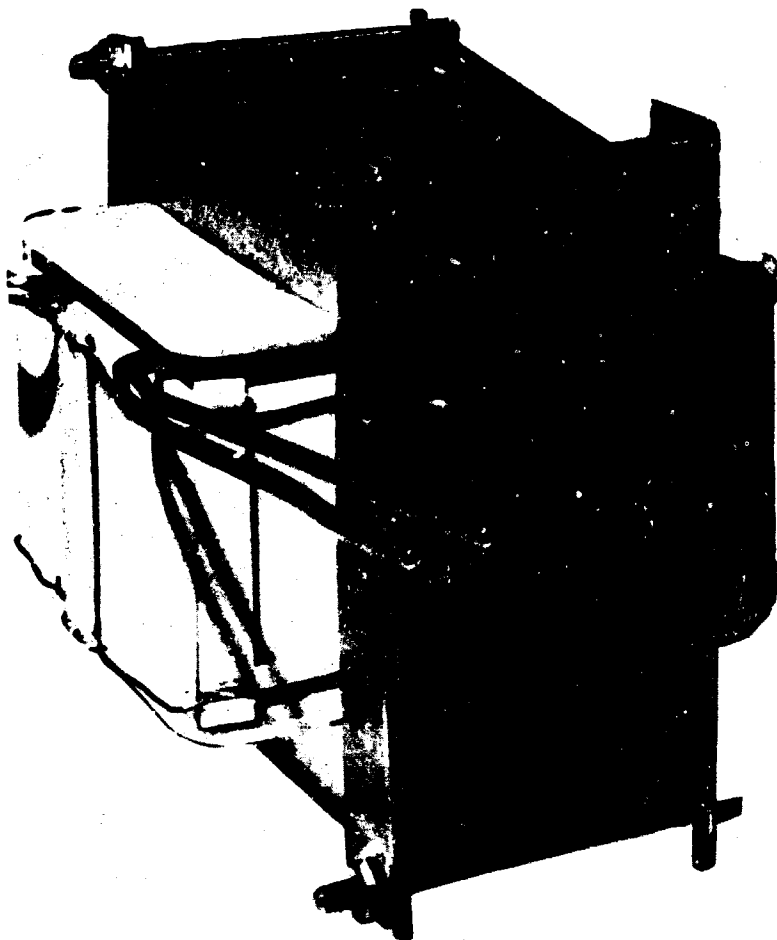


Figure 73. Continuous Windings Transformer

1/4-inch-thick end plates, all of type 316 stainless steel. Each container had four Eimac electrical feedthrough power terminals (two each for primary and secondary windings), thermocouple wells, potassium loading tube, and an Eimac sapphire window. The container for the transformer to be intentionally ionized also had accessories for potassium ionization, pressure measurement, and pressure control. Figures 74 and 75 show this latter test container.

The tube for potassium loading, evacuation, and sealing is shown in the foreground of figures 74 and 75. The closed portion of the tube was the potassium reservoir and prevented potassium from splashing on the transformer during loading. Potassium vapor entered the test container through holes in the tube. This type 316 stainless steel tube was TIG welded to the container top prior to final assembly. Thermocouple wells of thin wall type 316 stainless steel tubing were TIG welded in the test container tops. These wells were positioned so that thermocouples inside would indicate ambient, magnetic material, and conductor temperatures. Sapphire windows and electrical feedthrough terminals were also TIG welded in place. Sapphire windows allowed some visual observation of potassium phenomena. Electrical feedthrough terminals connected the transformer, inside the sealed potassium loaded test container, to conductors in the vacuum furnace.

The test container for ionization experiments contained accessories not on the other container. Two Eimac feedthrough terminals were mounted as shown in figure 75, and were used to initiate ionization. A stainless steel capacitance manometer head was TIG welded into the test container top and was used to determine test container pressure. Potassium pressure was controlled using the cold finger tubulation shown in figures 74 and 75. Potassium vapor pressure was thus a function of the coolest temperature available to the potassium. The cold finger was connected to a power feedthrough terminal on the water cooled furnace face plate by a heavy copper conductor. The power feedthrough acted as a heat sink when it was desired to lower test container pressure. The cold finger was heated by passing a current through it when it was desired to raise the pressure. An Inconel sheathed and isolated thermocouple was attached to the end of the cold finger to monitor cold finger temperature.

5. Assembly of Transformers in Test Containers

Transformers were prepared for attachment to test containers by routing leads to feedthrough rods, cutting excess length from leads, and plating lead ends. Lead end preparation and

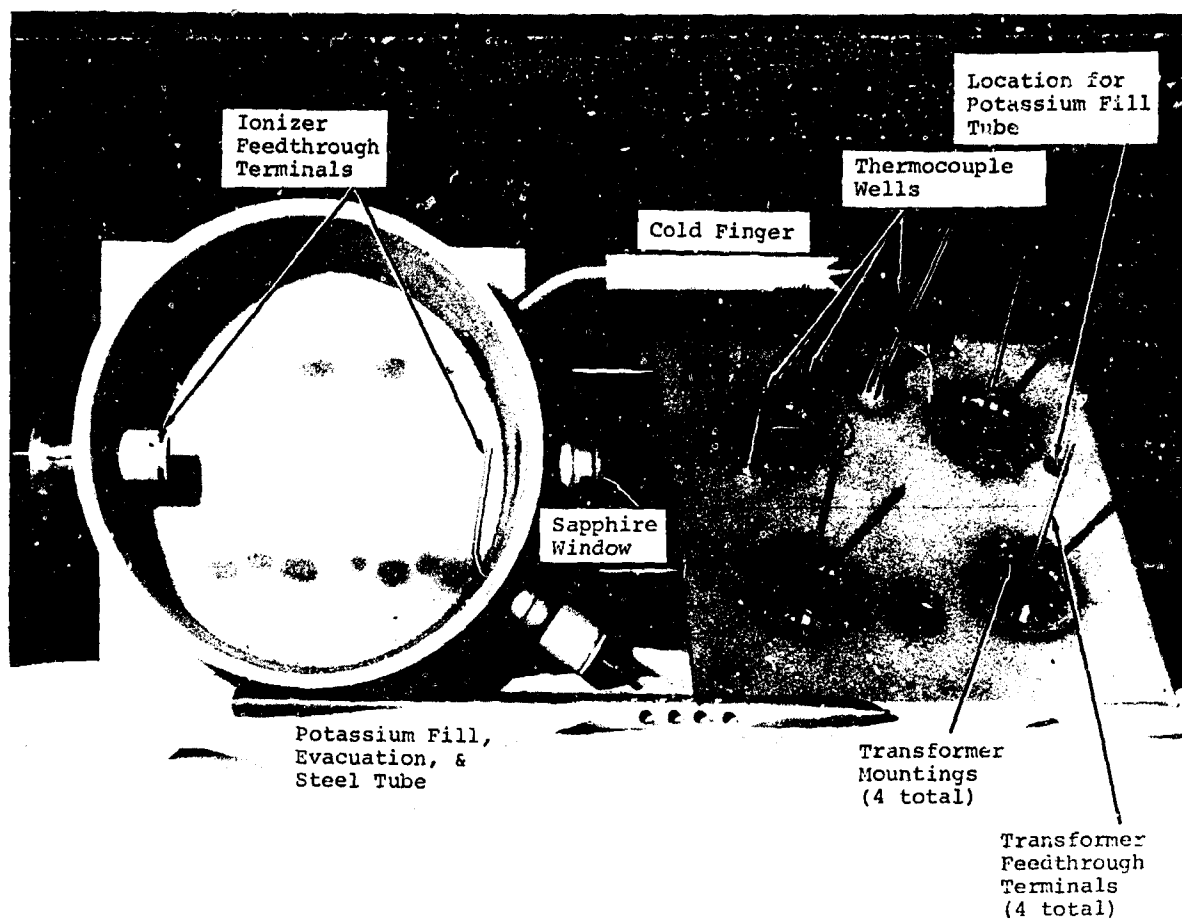


Figure 74. Container for Testing a Transformer in Ionized Potassium Vapor (Parts)

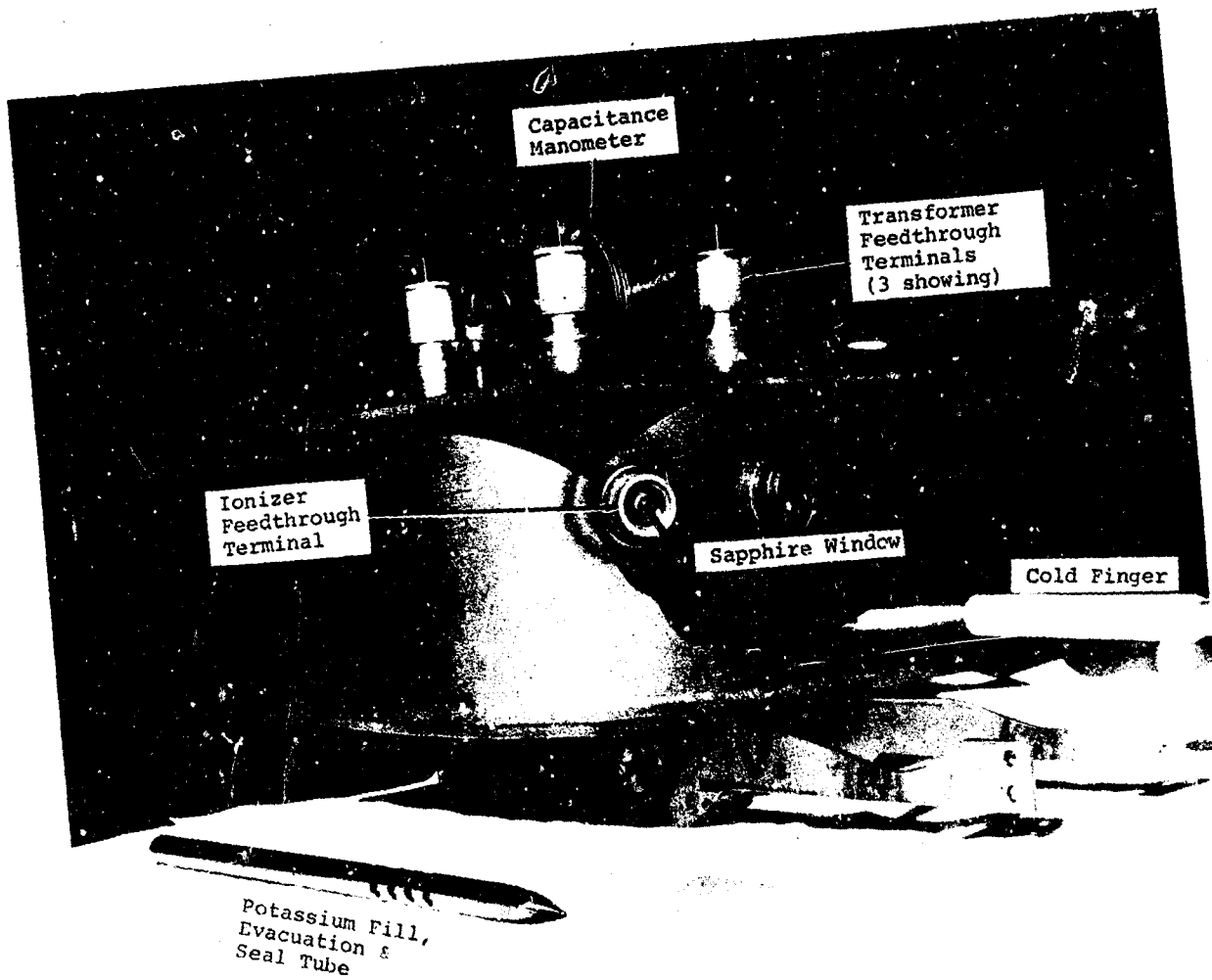


Figure 75. Container for Testing a Transformer in Ionized Potassium (Assembled)

plating were done as described earlier except that etching was done as for Inconel clad silver. The electrical terminal seal feedthrough rod to lead connections were made with a stainless steel connector. Transformers were mounted in test containers by attachment to container tops using mountings as shown in figure 74. Transformer tests without potassium were conducted with transformers mounted to container tops and tops tack welded to the remainder of the container. Potassium fill tubes were not in the container at these times.

After 600°C thermal vacuum tests without potassium, transformers were removed from test containers and examined. No degradation was found in either transformer. Potassium fill tubes were welded to test container lids as the first step toward potassium loading. The containers were leak checked with a CEC leak detector to determine if the thermal cycle had caused leaks. No leaks were found in either test container. Test containers were subsequently reassembled by welding the top in place, given final helium leak checks, and loaded with potassium. Potassium loading evacuation and sealing was done as described earlier with approximately 1.5 grams of potassium placed in the continuous winding transformer container and 2.5 grams of potassium in the joined winding transformer container.

6. Container for Visual and Electrical Tests in Ionized Potassium

A test container was built to study problems expected to be encountered in testing and evaluating transformers and ceramic-to-metal hermetic seals in ionized potassium vapor. This container was also used in tests designed to give data on potassium ionization phenomena specific objective of this container and its tests were to:

- a) Determine the suitability of a sapphire window exposed to 600°C potassium vapor to observe visually happenings inside test containers.
- b) Investigate the tendency for potassium to coat a sapphire window and electrical terminal seals and to evaluate the use of an external heat source to remove this potassium.
- c) Investigate the effects of a d-c field on ionization.
- d) Investigate the general phenomenon of potassium ionization.

Figure 76 shows this stainless steel test container which has an Eimac sapphire window and an Eimac feedthrough terminal. Figure 77 shows the container ready for tests. An electrical heater (shown in figure 76) was placed around the sapphire window and a coil of insulated conductor was placed around the feedthrough terminal (shown in figure 77). The center electrode of the feedthrough projected into the container and was unattached to the container. Approximately two grams of potassium were loaded into this container. Potassium loading, evacuation, and sealing processes used on this test container were the same as used on other potassium vapor test containers.

Potassium vapor coated the beryllia insulators of the feedthrough during room temperature storage, thus reducing the room-temperature terminal-to-container resistance from 10^{14} ohms before potassium to 10^{12} ohms as loaded. The potassium coatings on the beryllia increased in thickness as the test container was heated. Table XXIX shows the measured resistance-temperature relationships. After tests at 600°C , the room temperature resistance was approximately 2000 ohms.

The sapphire window became spotted with potassium when the test container was loaded. No other potassium became attached to the window during the initial room temperature tests. Potassium completely coated the window during 600°C tests thus preventing any visual observations at this temperature. Most of the potassium was removed from the window in cooling the test container to room temperature and visual observations could again be made.

Electrical breakdown or ionization of the potassium vapor between the feedthrough terminal and the remainder of the container was investigated. The power supply first used could not sustain ionization; therefore, each case of ionization was observed only briefly. Room temperature (26°C) ionization occurred at 540 volts d-c before the test container had been heated. The seal became coated with potassium at higher temperatures and no true vapor breakdown voltage could be obtained. Differences were noted in visual observations of the ionization pattern as the power supply polarity was changed. The positive ions cause a violet glow to occur at the cathode.

The effects of a magnetic field on ionized potassium were investigated. A coil consisting of 118 turns of 0.031-inch diameter Anadur insulated Inconel clad silver conductor was located coaxially around the ceramic portion of the electrical terminal seal. This coil was energized by a 20 volt, 10 amp, 60 Hz source. Tests were run with

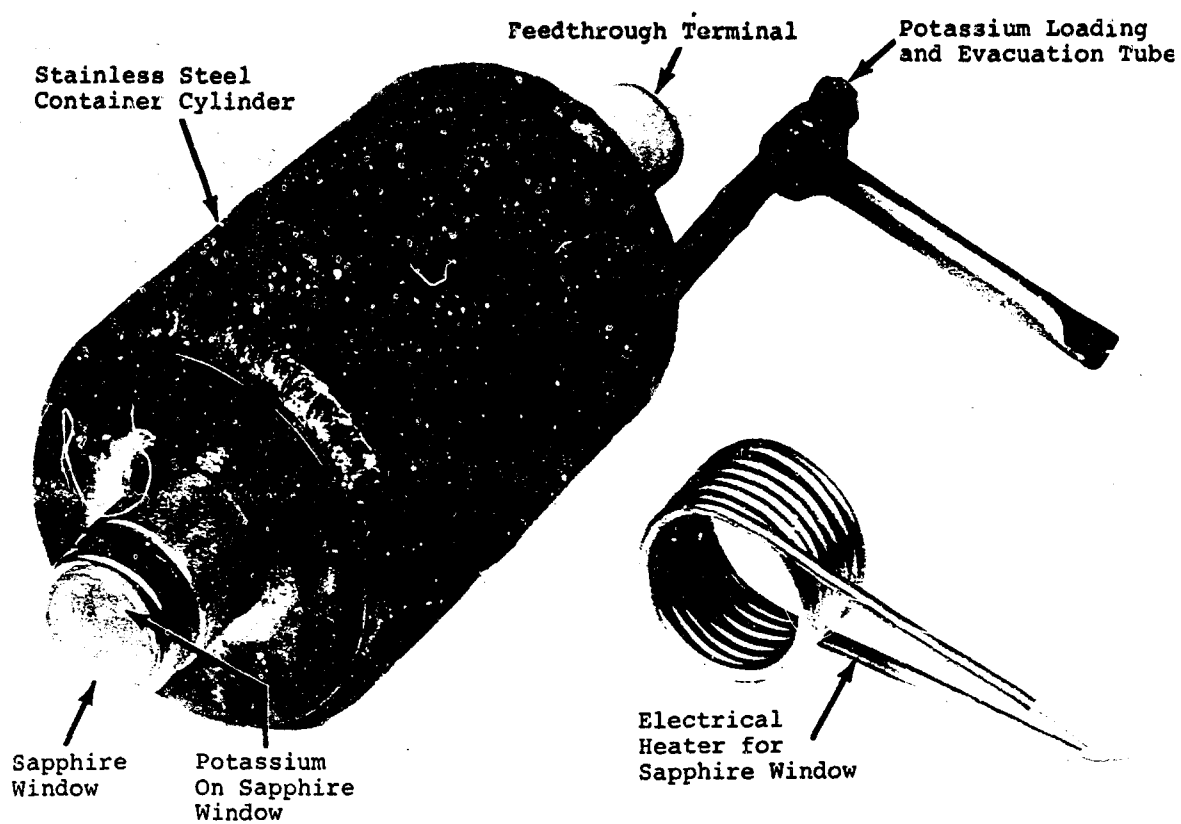


Figure 76. Window - Electrical Feedthrough Terminal Test Container

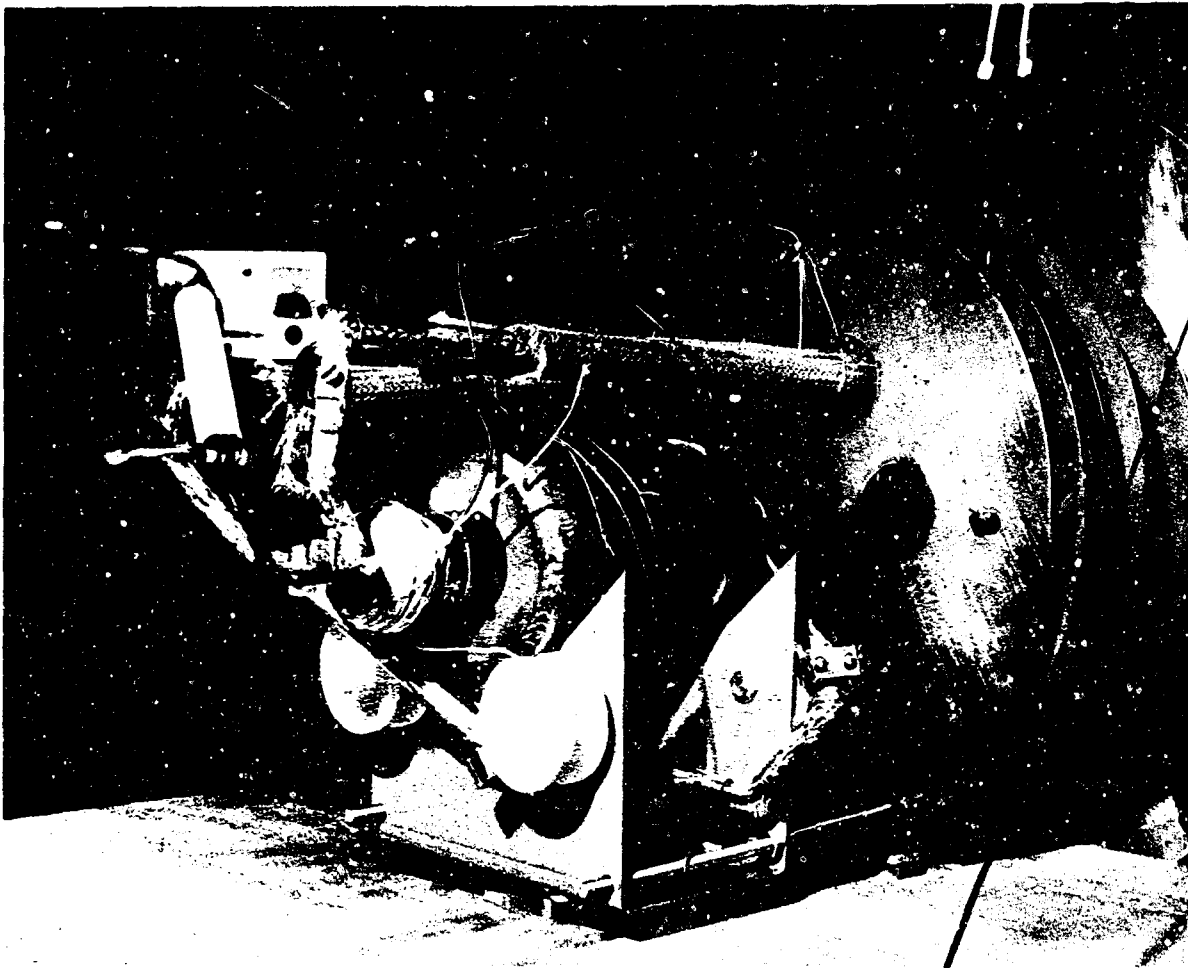


Figure 77. Sapphire Window - Electrical Terminal Seal Container
Ready for Test

TABLE XXIX. Resistance Measurements with Temperature Increase on BeO Terminal Seal Coated with Potassium

Instruments: Keithley Electrometer, Model 610B
Keithley Regulated High Voltage Supply, Model 240

Current (amp)	Voltage (volts)	Total Resistance ^(a) (ohms)	Temperatures (°C)		
			Feedthrough Terminal ^(b)	Case	Window
3×10^{-12}	100	3.33×10^{13}	26	24.5	24
2×10^{-11}	100	5.0×10^{12}	49	62	48
1.5×10^{-10}	100	6.68×10^{11}	78	97	71
1.6×10^{-9}	100	6.26×10^{10}	150	163	129
5.3×10^{-9}	100	1.89×10^{10}	211	213	193
2.5×10^{-8}	100	4.0×10^9	233	244	222
2.5×10^{-6}	100	4.0×10^7	327	330	301
5×10^{-4}	100	2.0×10^5	339	338	314
1.45×10^{-2}	100	6.9×10^3	497	497	475
1.25×10^{-1}	100	8.0×10^2	608	608	591

a - The area of BeO coated with potassium was 2.5 square inches with a length of one inch.
b - A thermocouple was against the BeO portion of the terminal seal.

the test container at 600°C and ionization (terminal to container) sustained at 57 volts, 1 ampere a-c. Ionization was quenched when the a-c power supply energized the coil. The terminal to container voltage rose to 590 volts as the circuit was broken.

Additional ionization testing was done after the test container was cooled to room temperature. The terminal to container resistance (across the beryllia) was approximately 2000 ohms. Sustained ionization was accomplished by impressing 220 volts a-c between terminal and container. The voltage across the coil which quenched ionization at 600°C would not quench ionization at room temperature.

One important difference between these experiments and the ionization experiments conducted on the four-terminal test container is the observed temperature changes. The four-terminal electrical resistivity test container temperature, as measured on the beryllia insulator of the electrical terminal seal, rapidly decreased when ionization occurred. The electrical terminal seal temperature in the window-feed-through test container increased when ionization occurred. It is not immediately apparent why this difference should be observed, unless the ion paths in the two containers were quite different and were not monitored in an identical manner by the fixed thermocouples.

7. Electrical Testing of 5-kVA Transformers

a. General Description of Test Procedures

The 5-kVA transformers, in their test containers, were placed inside a 10-kVA resistance heated hot wall vacuum furnace capable of 1×10^{-6} torr pressure at 600°C. The water-cooled face plate of the vacuum furnace was fitted with suitable high current (150 amps) vacuum feedthrough connectors. A pyrex window view port was also provided so potassium ionization around the transformer could be observed if the sapphire window on the transformer test container did not become coated with a thin film of potassium metal. Previous experiments had indicated this window might become coated.

Transformers were tested electrically at temperatures from 23°C to 600°C in the absence and presence of potassium to determine potassium vapor effects. The power supply and electrical load circuit used in transformer testing is shown in figure 78. Current and power meters were 0.25% accuracy type, and were made by either Weston or Westinghouse. Voltages were monitored on two electronic voltmeters. One voltmeter sensed average voltage and indicated average voltage times 1.11. The other sensed and indicated true rms voltages. A comparison of voltmeter readings gave a form factor value. An oscilloscope was used to observe waveforms.

The output power of the transformer was connected to no-load for open circuit testing, to a resistance load for power factor of 1.0 testing and a combination of inductive and resistance loads for a power factor of 0.75 lagging.

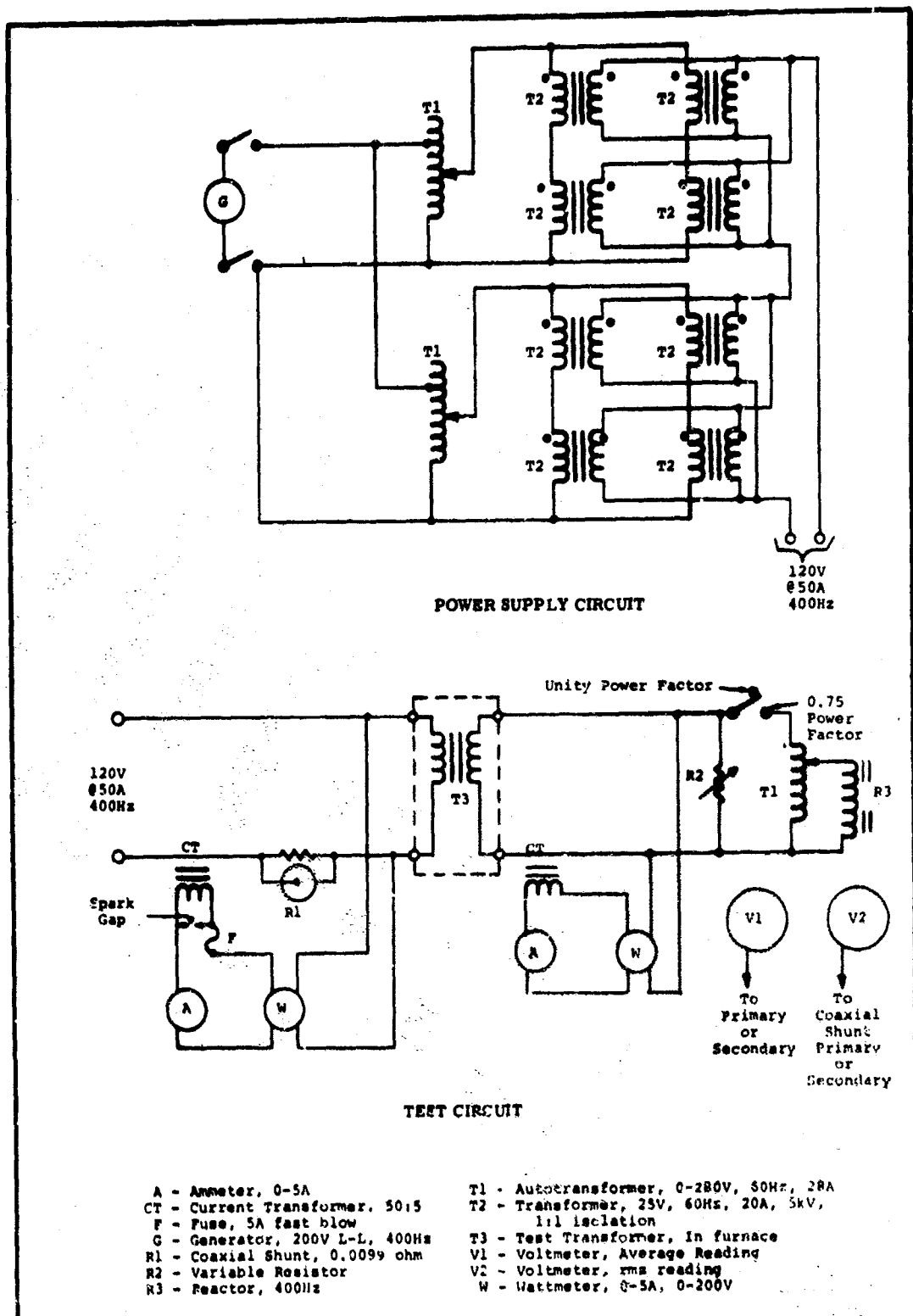


Figure 78. Transformer Test and Power Supply Circuits

b. Evaluation at 600°C With and Without Potassium

All transformer electrical tests were performed with the transformers in the hot-wall vacuum furnace. Tests without potassium were conducted with the transformer exposed to a 10^{-6} torr environment. Tests with potassium were conducted after the vacuum tests and with potassium sealed in test containers. Table XXX shows the electrical operating characteristics of the continuous winding transformer at various temperatures with and without potassium. The potassium vapor was not intentionally ionized, so this transformer operated in essentially a neutral potassium vapor environment. A comparison between two conditions, 591°C in vacuum and 555°C in potassium vapor, shows the efficiency to be the same in both cases; however, there appears to be better regulation with potassium present. The power factor changed from 1 to 0.91 with potassium. Otherwise, the electrical data presented in table XXX shows a little difference between the potassium vapor and 600°C thermal vacuum results.

The transformer temperature rise to equilibrium with and without potassium was conducted in a vacuum with identical load and power conditions. Temperature differences were obtained by subtracting the initial temperatures at the start of the experiment from the temperatures after certain elapsed time periods. The data obtained are shown in figure 79. This comparison indicates, when equilibrium is reached, the ambient temperature of the potassium is much lower than the transformer temperature. This is quite different from what one would expect as the transformer, being at a higher temperature, should transfer its energy to the potassium causing the vapor to be at approximately the same temperature as the transformer. In the cases where the ambient condition is a vacuum of approximately 1×10^{-5} torr, the ambient temperature around the transformer is approximately the same as the transformer core. If one could conceive of a mechanism whereby the thermal energy was conducted very efficiently to the furnace walls in the potassium experiments and not in the thermal vacuum, the differences could be explained. This is very difficult to do as the transformer and the container were identical in both cases.

After temperature rise data was taken, the furnace and transformer were energized. Both transformer losses and furnace heat were used to increase transformer temperature toward the 600°C test temperature.

TABLE XXX. Continuous Winding Transformer Thermal Test Data
With and Without Potassium

Description of Transformer Test	AC Volts In (Volts Av)	AC Volts In (Volts RMS)	Current In (amps)	Power In (watts)
Thermal Vacuum	120.0	121.0	5.53	153.6
Potassium Vapor*	120.0	124.0	4.24	164.0
Thermal Vacuum	9.28	9.3	50	160.0
Potassium Vapor*	7.93	8.2	41.7	120.0
Thermal Vacuum	120.0	120.0	46.3	5380
Potassium Vapor*	120.0	120.0	45.3	5400
Thermal Vacuum	120.0	121.5	47.8	4060
Potassium Vapor*	120.0	121.5	46.8	4060
Thermal Vacuum	120.0	118.0	47.5	5400
Potassium Vapor	120.0	120.5	45.8	5340
Thermal Vacuum	120.0	118.0	47.1	5400
Potassium*	120.0	120.5	46.2	5400
Thermal Vacuum	120.0	124.0	4.8	270
Thermal Vacuum	6.72	6.73	41.7	208

* Potassium Vapor was not ionized

st Data

t)	Power In (watts)	AC Volts Out (Volts Av)	AC Volts Out (Volts RMS)	Current Out (amps)	Power Out (watts)	Power Factor	Efficiency (percent)	Core Temp. (°C)
3	153.6	123.5	125.0	--	--	OPEN CIRCUIT	--	35
4	164.0	120.0	124.0	--	--	OPEN CIRCUIT	--	70
	160.0	0.247	--	--	--	SHORT CIRCUIT	--	43
	120.0	0.0137	--	--	--	SHORT CIRCUIT	--	84
	5380	119.5	119.5	42.2	4930	1	93.4	60
	5400	121.5	121.5	41.5	4960	1	93.5	101
	4060	116.0	118.0	42.0	3728	0.75	92.0	60
	4060	114.0	112.0	41.7	3720	0.75	92.0	101
	5400	119.0	119.0	43.8	5000	1	92.8	347
	5340	120.0	119.5	42.4	5000	1	93.9	362
	5400	114.0	115.0	43.7	5000	1	92.8	591
	5400	120.0	119.0	46.5	5000	0.91	92.8	555
	270	125.0	128.0	--	--	OPEN CIRCUIT	--	600
	208	--	--	--	--	SHORT CIRCUIT	--	569

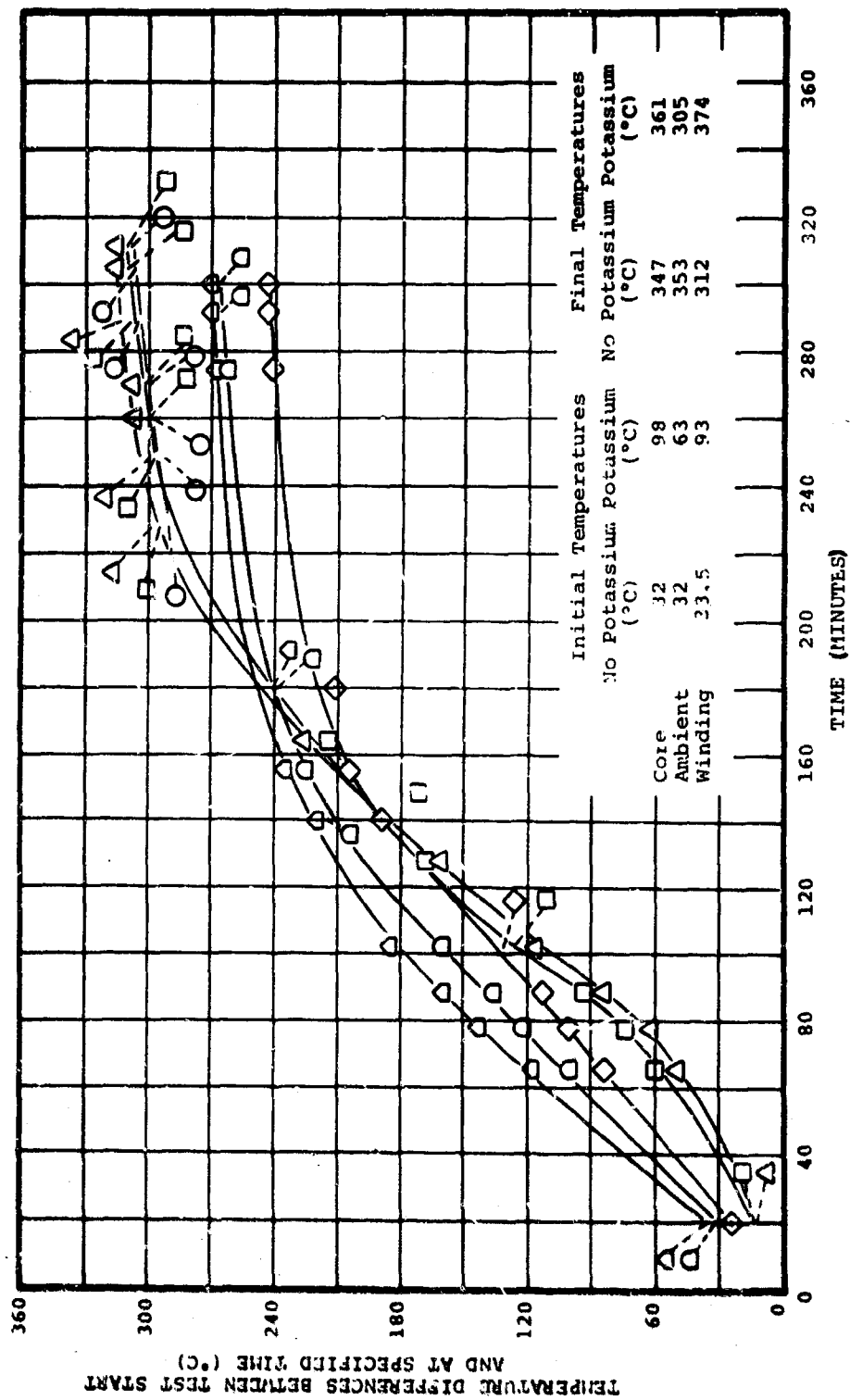


Figure 79. Temperature Rise of Transformer with and without Potassium Vapor
(Energized, 5 kVA Output at Unity Power Factor)

The furnace was fully on during this time period. The furnace was a type that had a saturable-core-reactor controller. This controller normally did not indicate a maximum output when the furnace was used in 600°C thermal vacuum testing. The presence of potassium vapor made it necessary to maintain a much higher temperature on the hot wall furnace to keep the transformer test temperature rising toward 600°C.

After considerable time, with the furnace energized at maximum capacity, a test temperature of 555°C was reached. Voltage and current readings were taken. These are also shown in table XXX. The current and voltage readings did not sag as did the previous thermal-vacuum readings. Approximately 2.5 hours after the 555°C readings the meter readings were observed to become quite erratic and an electrical discharge type noise was heard from the test container. Test temperature at this time is shown in table XXXI 193 minutes after the furnace was turned on. Electrical meter readings fell to zero and it was discovered the fuses had blown. The transformer primary and secondary windings were checked and both were found to be open.

The potassium in the test container was removed, and the container was cut open by use of a metal band saw. Figure 80 shows the appearance of the transformer. Top windings were vaporized and the nickel clad silver deposited randomly over the inside of the test container and other parts of the transformer.

c. Evaluation at 600°C With and Without Ionized Potassium

A second transformer was fabricated by joining the windings instead of a continuous winding. Table XXXII shows the data obtained when the transformer was operated at temperatures to 600°C with and without potassium.

The transformer could not be energized in potassium vapor without destructive ionization above 100 volts a-c. Above this voltage high current low voltage readings indicated an ionization conduction path from terminal seal to ground. The voltage input was therefore kept below or at 80 volts a-c in the majority of electrical tests.

TABLE XXXI. Transformer Temperature

Time (min)	Thermal- couple No. 1 Core	Thermal- couple No. 2 Ambient	Thermal- couple No. 3 Top of Core	Thermal- couple No. 4 Ambient	Thermal- couple No. 5 Middle Winding	Thermal- couple No. 6 Top Winding
Start	376	325	388	338	38	369
25	421	403	434	405	425	419
50	482	499	500	490	498	500
65	526	531	542	531	532	532
95	542	543	582	569	576	576
160	555	544	571	550	566	566
163			577		573	573
166			582		582	577
169			588		582	582
172			593		585	585
174			593		593	593
176			599		596	596
178			599		599	599
182			599		582	582
184			597		582	582
186			596		588	588
188			599		596	596
190			597		596	596
193			599		596	599
Transformer Failed						
196			587		577	577
199			577		571	571
202			571		565	565
205			588		571	566
208			582		571	571
211			582		571	571
214			582		571	571
NOTE: After 160 minutes, the temperatures of the transformer at Thermo- couple Nos. 3, 5, and 6 were recorded on a Brown Electronic recorder. Other temperatures were taken on a Leeds and Northrup Potentiometer manually.						

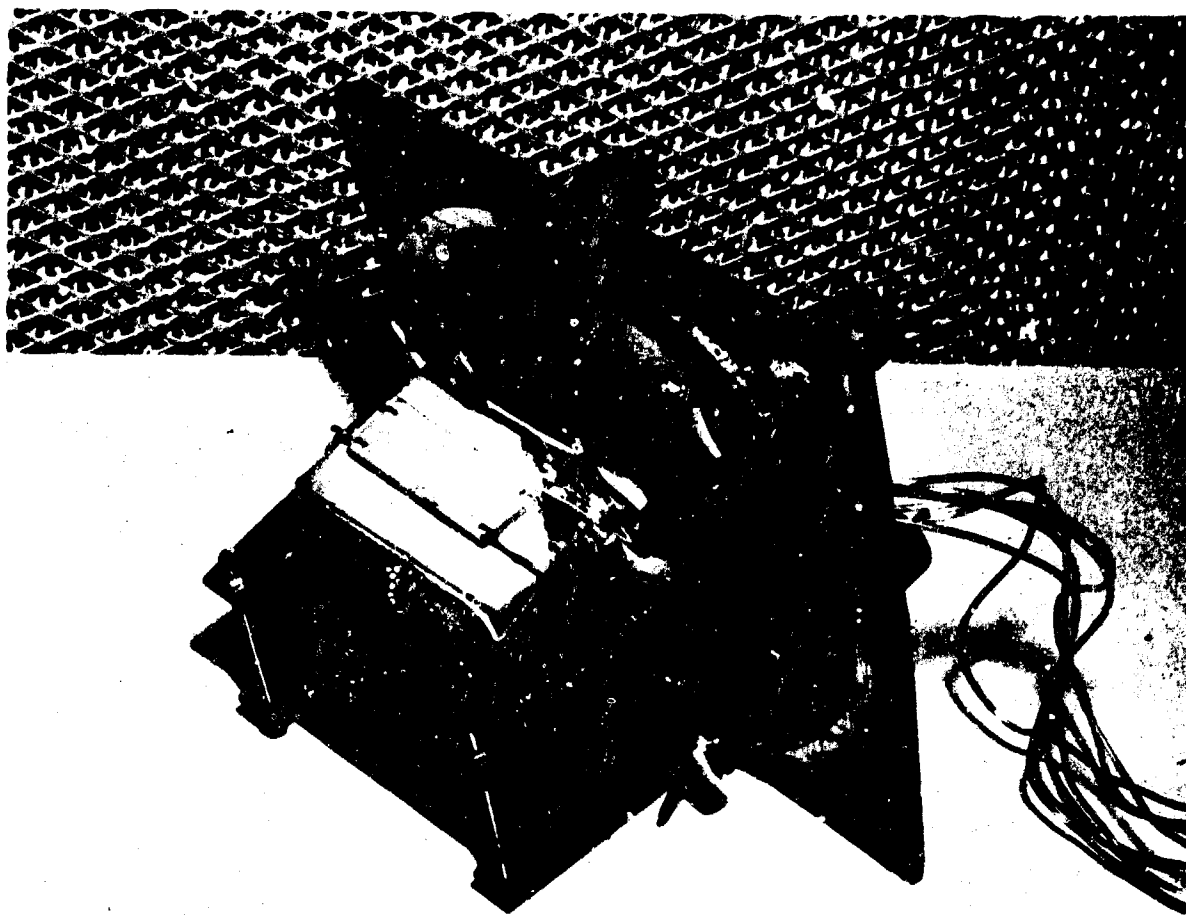


Figure 80. Continuous Winding Transformer After Failure

TABLE XXXII. Joined Winding Transformer Thermal Test Data
With and Without Ionized Potassium

Description of Transformer Test	AC Volts In (Volts Av)	AC Volts In (Volts RMS)	Current In (amps)	Power In (watts)
Thermal Vacuum	120.0	124.0	8.8	196
Ionized Potassium Vapor	101.0	100.0	3.6	130
Ionized Potassium Vapor	109.0	112.0	4.9	148
Thermal Vacuum	120.0	118.0	51.0	4080
Ionized Potassium Vapor	80.5	79.5	47.8	3660
Thermal Vacuum	120.0	120.0	46.4	5360
Ionized Potassium Vapor	80.0	80.0	46.5	2840
Thermal Vacuum	120.0	122.0	47.2	5400
Ionized Potassium Vapor	40.0	39.5	25.2	720
Ionized Potassium Vapor	62.0	60.3	25.5	1750
Thermal Vacuum	4.95	4.94	30.0	120
Thermal Vacuum	120.0	126.0	12.5	84
Neutral Potassium Vapor	30.0	30.0	40.8	2040

Test Data
n

Current In (amps)	Power In (watts)	AC Volts Out (Volts Av)	AC Volts Out (Volts RMS)	Current Out (amps)	Power Out (watts)	Power Factor	Efficiency (percent)	Core Temp. (°C)
8.8	196	129.0	132.0	--	--	OPEN CIRCUIT	--	35
3.6	130	107.0	109.0	--	--	OPEN CIRCUIT	--	38
4.9	148	116.0	118.0	--	--	OPEN CIRCUIT	--	42
51.0	4080	121.0	124.0	41.8	3700	0.71	90.7	23
47.8	3660	80.0	79.8	42.9	3300	0.97	91.5	23
46.4	5360	125.0	125.0	40.2	5000	1.0	93.2	352
46.5	2840	75.0	73.0	41.9	2510	0.82	88.5	419
47.2	5400	121.5	122.0	40.8	5000	1.0	92.6	600
25.2	720	37.9	37.5	20.7	620	0.79	86.4	564
25.5	1750	60.0	60.0	32.5	1520	0.79	89.9	550
30.0	120	0.0439	0.0457	--	--	SHORT CIRCUIT	--	600
12.5	84	128.0	135.0	--	--	OPEN CIRCUIT	--	600
40.8	2040	36.0	36.0	35.0	1760	0.75	86.5	600

Potassium vapor close to the transformer was ionized by impressing 170 volts a-c between the ionization terminal seal and the test container. This ionization did not appear to affect the operation of the transformer. At room temperature ($\sim 23^{\circ}\text{C}$), a glow discharge from this ionization of potassium vapor was observed through the sapphire window to fluctuate in intensity with sudden intense bursts of light. The same observations could not be made at elevated temperatures as the window became coated with a reflective coating of potassium.

This transformer was raised to 600°C in a de-energized condition by setting the hot wall vacuum furnace controller at 800°C . The core, top of the winding, ambient of potassium vapor, and the cold finger temperatures were respectively 607° , 595° , 591° , and 268°C before the transformer was energized. The transformer was energized with 30 volts a-c and 40.8 amperes. The input and output of the transformer are shown in the last row of data in table XXXII. During the time these electrical data were taken, oscilloscope traces indicated drastic wave form changes in the transformer input and output voltages at random time intervals. The voltage waveforms observed had a d-c component that apparently caused some very unusual electrical meter indications. The indicated apparent power in watts of both the transformer input and output was much less than the true watts obtained from the wattmeter. The voltmeters used respond only to alternating sine waveforms, therefore, the distorted waveforms observed might explain the meter readings.

In contrast to the usual temperature over-shoot of this vacuum furnace when a temperature of 600°C was reached, the thermocouple readings immediately after the transformer was energized fell rapidly. The temperatures corresponding to the thermocouple readings, after 5 to 10 minutes of transformer operations in the saturated potassium vapor were; transformer core 508°C , windings 502°C , potassium vapor 508°C , and the cold finger 280°C . The pyrometer on the furnace controller indicated the temperature of the whole furnace mass was also decreasing rapidly. This condition continued until the 30-amp fuse in the input power supply interrupted the circuit. The distorted waveform with d-c components could cause the current transformer to saturate. A saturated current transformer would not accurately transmit a sine wave signal to the meters and the test data could be in error.

This would explain the electrical results obtained, however, the thermocouple readings in both transformer tests show a real temperature decrease and these phenomena cannot be explained by known theory.

If the electron in the outermost shell of a large number of the potassium atoms could be placed in some excited state or states, not ionized, the energy required to do this might be on the same order of magnitude as the temperature drop of the potassium vapor in these experiments. This condition, however, is very unlikely and more work in this area would need to be done to generate proper data to prove this assumption.

8. Examination of Transformer Components After Exposure to 600°C Potassium

Both transformer failures were mainly in the windings. Primary and secondary winding leads were burned open near where the windings started. Silver and nickel from the vaporization of portions of these leads was found on transformer parts and on the inside of test containers. Secondary and primary windings were melted in several places as is shown in figure 81. Portions of the Hiperco 27 alloy

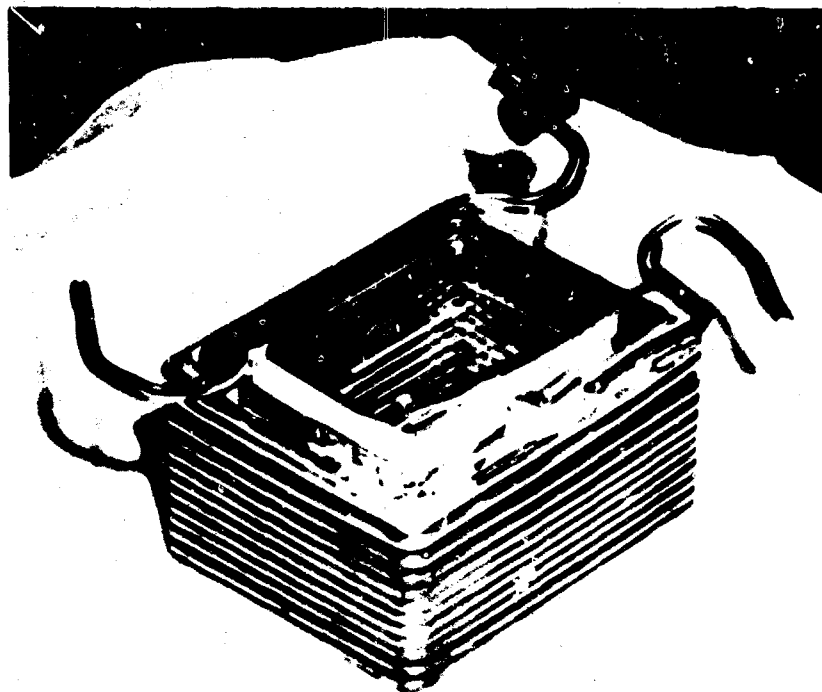


Figure 81. Windings From a Failed Transformer

cores were vaporized and several laminations adjacent to these places were fused together. Some of the stainless steel parts were melted or vaporized. Transformer failures occurred in a short time and apparently involved a considerable amount of energy. Figure 82 shows the joined winding transformer after tests.

A specimen of nickel clad silver conductor was taken from the continuous winding transformer near a place where the conductor had melted. Figure 83 shows this conductor. Considerable grain growth can be seen by comparing this structure with that from the 5000 hour 600°C potassium exposed material, figure 14. Grain growth resulted from temperatures experienced at the time of failure. No evidence of potassium effects can be found.

Transformer laminations were examined for possible erosion as was found on the Rowland rings (see figure 43). Figure 84 shows erosion did take place. Again erosion was on the side opposite that having plasma-arc sprayed alumina interlaminar insulation. Transformer tests combined magnetic field and potassium, and caused, in a short time, more erosion of Hiperco 27 alloy than did 5000-hours in 600°C saturated potassium vapor.

The ceramic materials in the transformer consisted of single crystal sapphire mat, Alite A-612 and A-610 turn-to-turn spacers, and Alite A-610 coil form parts. Alite A-612 was used to complete this transformer as additional Alite A-610 was not readily available. The A-610 (99% Al_2O_3 1% MgO) and the sapphire mat were not adversely affected by potassium. The A-612 ceramic (containing approximately 3.4% silica) retained their form but could be easily crumbled to powder. This was not unexpected and did not contribute to the transformer failure.

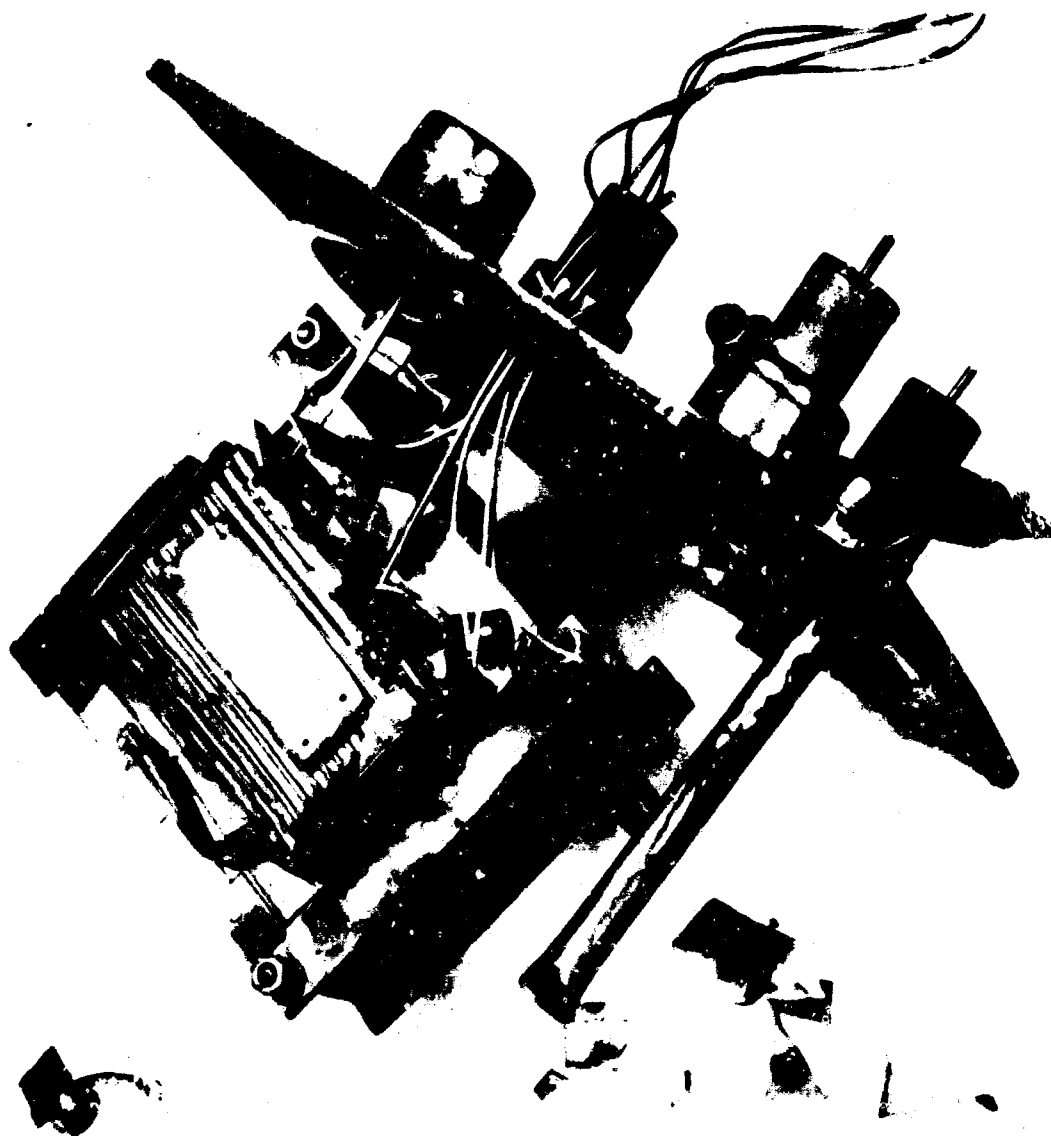


Figure 82. Joined Winding Transformer After Failure



Figure 83. Micrograph of Transformer Conductor After 600°C Potassium Vapor Exposure (100X)

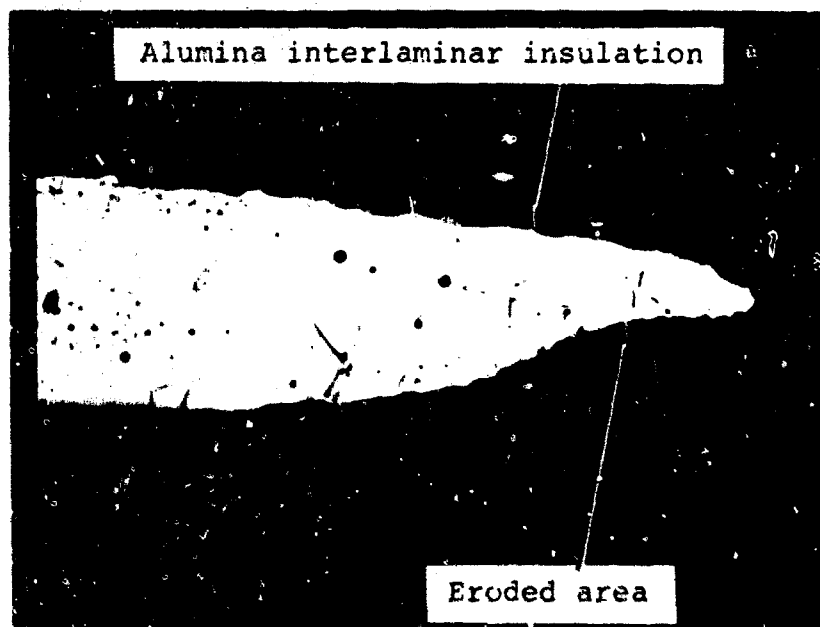


Figure 84. Micrograph of Transformer Lamination After Exposure to 600°C Potassium Vapor Exposure (200X)

SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

- 1) Very pure rhodium and iridium wires show excellent resistance to the corrosive action of 600°C potassium vapor.
- 2) Nickel-clad and Inconel-clad silver core conductors are resistant to 600°C potassium vapor and do not exhibit a large decrease in yield strength.
- 3) Cubex alloy and Hiperco 27 alloy show good resistance to 600°C potassium vapor. Hiperco 27 alloy becomes equally brittle after either potassium or thermal vacuum exposure, but exhibits no difference between thermal vacuum and thermal vacuum plus potassium vapor exposure.
- 4) Beryllia (99.8%), Coors AD999, aluminum oxide-yttrium oxide eutectic composition, and yttrium oxide are electrical insulating materials that can be used in electrical devices exposed to 600°C potassium vapor for 5000 hours with essentially no degradation.
- 5) Alumina, plasma-arc sprayed on magnetic laminations in an inert atmosphere, can be applied 0.3 mils thick to give a potassium vapor resistant interlaminar insulation.
- 6) An electrical terminal seal of high purity beryllia can be fabricated that is resistant to 600°C potassium vapor for 900 hours.
- 7) A high temperature Rowland ring core box can be constructed to determine the performance of magnetic materials in potassium vapor at temperatures to 238°C.
- 8) Potassium vapor coats all ceramic materials at 450°C and reduces their usefulness as electrical insulators.
- 9) The outside of a tubular beryllia terminal seal does not become coated with potassium to the same extent as the inside; therefore, all electrical measurements conducted in potassium vapor should be conducted with inverted electrical terminal seals.

- 10) The electrical resistivity of a conductor can be continuously measured in the presence of 600°C potassium vapor.
- 11) Saturated potassium vapor in an alternating magnetic field eroded the magnetic core material of two test devices exposed to it.
- 12) Operation of a transformer in ionized potassium vapor using voltages in excess of 100 volts at 600°C was not possible because of short circuiting through the potassium ion vapor.
- 13) Temperature changes were observed during certain time periods when a transformer was operated in a potassium vapor environment at elevated temperatures. These temperature changes cannot be explained by known conventional theory.

B. RECOMMENDATIONS

Electrical devices operating in potassium vapor encounter combined effects of potassium vapor and electric and magnetic fields. Potassium vapor exposure alone does not give sufficient design information for these applications. Additional work needs to be undertaken in areas combining potassium vapor with magnetic and electric fields. Fundamentals of the interactions of potassium and electric and magnetic fields, and the effects of these on materials and device design, need to be understood. When a better understanding of the above phenomena is gained, small scale devices (e.g., motors or generators) should be built to investigate the added effects due to rotating members.

The unexplained temperature decrease in an electrical device operating in 600° to 850°C potassium vapor should be investigated to determine the cause of this phenomenon. If it can be established that the potassium vapor surrounding an operating transformer is much lower in temperature than the transformer, a calorimetric heat exchanger should be designed and constructed to see if a practical cooling device could result from this effect. Such a device would have many industrial and aerospace applications.

APPENDIX I

CALCULATION OF MECHANICAL PROPERTIES

Modulus-of-rupture strengths were calculated from specimen dimensions, breaking forces, and dimensions from the four point loading fixture shown in figure 8. The following formula was used:

$$MOR = \frac{3(L-l)P}{2ab^2} ; \text{ where:}$$

MOR = Modulus-of-rupture strength (psi)

L = Fixture length = 0.80 inch

l = Fixture length = 0.25 inch

a = Measured specimen width (inch)

b = Measured specimen height (inch)

P = Breaking force (lbs)

Yield and 0.2% offset yield strengths were determined according to the following formula:

$$S = \frac{P}{A}; \text{ where:}$$

S = Strength (yield or 0.2% offset in psi)

P = Force at yield or 0.2% offset (lbs)

A = Area of specimen cross section (inch²)

APPENDIX II

STATISTICAL ANALYSES

Mean or average values from mechanical test data were determined by the following formula⁹:

$$M = \frac{\sum X}{N}$$

Where M = Mean or Average Value
X = Numerical Value
N = Total number of values

Standard Deviation was calculated by the following formula:

$$S.D. = \sqrt{\frac{\sum (X-M)^2}{N}}$$

Where S.D. = Standard Deviation

Coefficient of Variation in percent was calculated by the following formula:

$$C.V. = \frac{S.D.}{M} \times 100$$

Where C.V. = Coefficient of Variation (%)

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